QUALITY ASSURANCE PROJECT PLAN

for Soil Removal at the Old American Zinc Site

Fairmont City, Illinois

Prepared by:

ENTACT

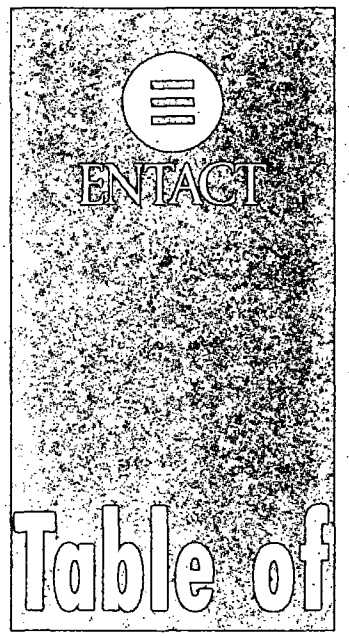

Table of contents

Quality Assurance Project Plan, Revision 1
Commercial, Industrial and Residential Removal Action for the
Old American Zinc Site
Fairmont City, Illinois

TABLE OF CONTENTS

Quality Assurance Project Plan, Revision 1
Commercial, Industrial and Residential Removal Action for the
Old American Zinc Site
Fairmont City, Illinois

TABLE OF CONTENTS

Quality Assurance Project Plan, Revision 1
Commercial, Industrial and Residential Removal Action for the
Old American Zinc Site
Fairmont City, Illinois

TABLE OF CONTENTS

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Commercial, Industrial and Residential Removal Action for the
Old American Zinc Site
Fairmont City, Illinois

TABLE OF CONTENTS

LIST OF ACRONYMS/ABBREVIATIONS

AOC	Administrative Order on Consent
ARARs	Applicable or Relevant and Appropriate Requirements
ASTM	American Standards for Testing Materials
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act (Superfund)
COC	Chain of Custody
CLP	Contract Laboratory Program
DQO	Data Quality Objective
EMSL	Environmental Monitoring and Support Laboratory
HASP	Health and Safety Plan
IEPA	Illinois Environmental Protection Agency
ICP	Inductively Coupled Plasma
ICVS	Initial Calibration Verification Standard
ISA	Integrated Site Assessment
MS/MSD	Matrix Spike/ Matrix Spike Duplicate
mg/kg	Milligrams/kilograms
NIST	National Institute of Standard Technology
NPL	National Priorities List
OSC	On-Scene Coordinator
PARCC	Precision, Accuracy, Representativeness, Completeness, Comparability
PPE	Personal Protective Equipment
PCBs	Polychlorinated Biphenyls
QA/QC	Quality Assurance/ Quality Control
QAPP	Quality Assurance Project Plan
RA	Removal Action
RPD	Relative Percent Differences
RAS	Routine Analytical Services
RCRA	Resource Conservation and Recovery Act
RI/FS	Remedial Investigation/ Feasibility Study
RD/RA	Remedial Design/ Remedial Action
RPM	Remedial Project Manager
SARA	Superfund Amendments and Reauthorization Act
SMC	Sample Management Coordinator
SOP	Standard Operating Procedure
SRM	Standard Reference Materials
SOW	Statement of Work
STL	Severn Trent Laboratory
SVOC	Semi Volatile organic Compounds
SW846	Test Methods for Evaluating Solid Waste 1986.
TAL	Target Analyze List
TCL	Target Compound List
TCLP	Toxicity Characteristic Leaching Procedure
TPH	Total Petroleum Hydrocarbon
TSP	Total Suspended Particulate Matter
USEPA	United States Environmental Protection Agency

VOA	Volatile Organic Analysis
VOC	Volatile Organic Compound
XRF	X-Ray Fluorescence

ENTACT
Section
one

1.0 PROJECT DESCRIPTION

1.1 INTRODUCTION

This Quality Assurance Project Plan (QAPP) has been developed by ENTACT & Associates, LLC (ENTACT) for the Commercial, Industrial and Residential Yard Soil Removal Action, Old American Zinc Site (Site) for use in conjunction with the Work Plan and Health and Safety Plan (HASP). These are distinct documents that form the project operations plan intended to guide field personnel, contractors, and other involved parties in all aspects of field operations. This QAPP will provide quality assurance and quality control (QA/QC) procedures for activities conducted during the off-Site sampling investigation and removal action (RA) performed in accordance with the Administrative Order on Consent (AOC) for the Site located near Fairmont City, St. Clair County, Illinois.

The United States Environmental Protection Agency (EPA) policy requires that all remedial activities be under the control of a centrally managed QA program. This requirement applies to all environmental monitoring activities supported by the EPA. Each contractor that generates data has full responsibility to implement minimum procedures to ensure that precision, accuracy, representativeness, completeness, and comparability (PARCC) of these data are known. To meet this objective, this Site-specific QAPP has been prepared detailing QA/QC procedures to ensure data generated during the remedial activities are accurate, precise, comparable and complete and therefore, representative of Site conditions.

This QAPP will serve as a controlling mechanism during the performance of the sampling and analysis activities to detail procedures to ensure that technical data gathered during the investigation and removal phase are accurate, precise, complete, and representative of actual field conditions and meet minimum requirements of the design. All QA/QC procedures will be structured in accordance with applicable technical standards, EPA requirements, and regulations in general accordance with the EPA Region 5 Model QAPP guidelines under the Resource Conservation and Recovery Act (RCRA).

1.2 SITE/FACILITY DESCRIPTION

1.2.1 Location

The Site is an approximately 132 acre parcel of land located at 2575 Kings Highway near Fairmont City, Illinois in the southeast quarter of Section 4, Township 2 North, Range 9 West in St. Clair County, Illinois. The Site includes a former zinc smelting facility.

The area around the Site is a mixture of commercial, industrial and residential properties. The Site is currently owned by XTRA Intermodal and is being used for semi-trailer storage.

1.3 SITE/FACILITY HISTORY

1.3.1 General History

In 1916, American Zinc Company, a subsidiary of American Zinc, Lead and Smelting Company, purchased an existing smelter from Granby Mining and Smelting. American Zinc operated the smelter until 1967 when it was decommissioned and the facility removed.

In 1976, the Site was leased to XTRA Intermodal, Inc. (XTRA) for the storage of semi-trailers. In 1979 XTRA purchased the property from Azcon Corporation, a successor to American Zinc, and XTRA is the current owner. The Site is currently being used for semi-trailer storage.

Currently, the only remnants of the former smelter operation are some accumulations of slag and demolition debris located along the northern portion of the Site. Former slag piles that were present on the Site were ground by XTRA and used to surface areas of the Site and provide a level surface. In the late 1990's in response to concerns expressed by the regulatory agencies. XTRA covered the area previously surfaced with ground slag with an asphalt product. An eight-foot chain link fence with locking gates controls access to the Site.

1.3.2 Past Regulatory and Data Collection Activities

In November 1994, the Illinois Environmental Protection Agency (IEPA) conducted an Integrated Site Assessment (ISA) at the Site. As part of the ISA, the IEPA sampled soils from the Site and 15 properties surrounding the Site and found the concentration of several metals above normal background levels. These metals included zinc, lead, cadmium and iron. Lead was the metal of greatest concern relative to potential human health risks. The other detected metals were generally found at the same locations as those exhibiting elevated lead concentrations.

Approximately half of the 15 properties sampled by IEPA had soil lead concentrations in exceedence of the EPA 400 milligrams/kilogram (mg/kg) residential action level for lead. Two of the properties exhibited soil lead concentration in exceedence of the EPA 1,000 mg/kg commercial/industrial action level for lead. The highest recorded off-Site soil lead concentration was 1,230 mg/kg.

Soil samples were collected from properties located adjacent to the Site in all directions. The residential action level for lead was exceeded in all directions with the exception of the properties located to the east of the Site. Although the majority of the lead exceedences were recorded on properties adjacent to the Site, properties up to five blocks from the Site showed lead concentrations in exceedence of the residential action level.

In November 1999, the EPA re-sampled the Site and several of the adjacent residential properties that were previously sampled by the IEPA and confirmed the presence of elevated metals concentrations in the soils. To date, no systematic sampling has been conducted to define the extent of possible off-Site impact associated with the past smelter activities.

1.3.3 Current Status

Based on the findings of the IEPA and EPA, an AOC was entered into between the EPA and American Zinc (the Respondents). The AOC requires the Respondents to conduct a removal action (RA) in connection with the property to abate an imminent and substantial endangerment to the public health, welfare or the environment that may be presented by the actual or threatened release of hazardous substance at or from the Site.

1.4 PROJECT OBJECTIVES AND INTENDED DATA USAGES

The objectives of the project are to address potential imminent and substantial endangerment to public

health, welfare or the environment in commercial, industrial and residential properties located in all directions from the Site. In addition, the potential for releases from the Site to re-contaminate off-Site properties will be evaluated. In accordance with the AOC and the Work Plan (Attachment 1 to the AOC), the following tasks are to be completed to meet these objectives:

- Preparation of a Site specific QAPP and HASP;
- Perform systematic off-Site soil sampling of residential, industrial and commercial properties surrounding the Site in all directions;
- Conduct soil removal on properties or portions of properties which are found to have soil lead concentrations above the action levels of 400 mg/kg for residential use and 1,000 mg/kg for industrial/commercial use; and,
- Evaluate and control possible recontamination of surrounding properties due to air transport and/or surface water runoff from the Site.

The purpose of the data to be generated as part of the off-Site sampling event and subsequent RA as covered under this QAPP is to define the extent of impacts to the surrounding properties and to verify that the performance standards for all associated tasks have been met. In addition, sufficient data will be gathered during project activities to verify that the performance standards associated with the short-term implementation of the RA (i.e., air deposition sampling, waste characterization sampling, sampling of backfill material etc.) are met.

Data collected as part of the RA will need to meet the Data Quality Objectives (DQOs) applicable for the end use of the data that was collected. As such, different data uses may require different levels of data quality. DQOs are qualitative and quantitative statements that specify the quality of results required to support decisions made during the project and have been developed in accordance with the Quality Objectives Interim Guidance Document (EPA QA/G-4).

The three types of DQOs identified for use at the Site include the following:

- Screening (DQO Level 1): This provides the lowest data quality but the most rapid results. It will be used for field screening and health and safety monitoring and preliminary comparison to action levels. This type of data will be used for personal air monitoring equipment as described in the Site specific HASP.
- Engineering (DQO Level 3): This provides an intermediate level of data quality and is used for Site characterization. Engineering analyses will include X-Ray fluorescence (XRF) instrument screening and laboratory data with quick turnaround times used for field screening but without full QC documentation. This type of data will be used for XRF screening for soil characterization on the properties, backfill characterization, and waste characterization.
- Confirmational (DQO Level 4): This provides the highest level of data quality and is used for purposes of risk assessment and evaluation of remedial alternatives. This requires full analytical and

data validation procedures in accordance with EPA recognized protocol. This type of data will be used for all off-Site soil sampling and laboratory analysis to confirm XRF data.

1.4.1 Project Target Parameters

A summary of the project tasks, the associated sampling parameters and the intended data usage are presented in Section 1, Table QAPP-1. Specific tasks are described in the following sections.

1.4.1.1 Off-Site Pre-Excavation Soil Sampling

An off-Site-sampling event will be conducted to define the extent of properties surrounding the Site that have soil lead concentrations in exceedence of the EPA RA levels. The RA level for soil lead concentrations for residential and industrial/commercial properties are 400 mg/kg and 1,000 mg/kg respectively. Soil sampling and analysis will be conducted in accordance with the AOC, Work Plan and approved QAPP.

Residential properties will be evaluated by collecting composite samples from the front yard, back yard and the drip zone around the perimeter of the house at the three specified depths of 0-6 inches, 6-12 inches, and 12-18 inches. All samples will be analyzed in the field for arsenic, lead, zinc, and cadmium using XRF equipment. Ten percent of the XRF samples will be sent to an off-Site laboratory for total RCRA metals and zinc analysis. The amount of composite samples collected from the residential properties for XRF analysis will range from seven composite samples collected from properties less than 10,000 square feet in size to thirteen composite samples collected from properties greater than 10,000 square feet in size.

Commercial/industrial properties will be evaluated using the same protocol as specified for the residential properties. The amount of samples collected on the commercial/industrial properties will range from six composite samples collected from properties less than 20,000 square feet in size to twelve composite samples collected from properties greater than 20,000 square feet in size.

Once it is determined that the potential lead impacts are surficial, composite sampling will be limited to the 0-6 inch depth interval for both the residential and commercial/industrial properties.

If lead is found in concentrations exceeding the RA levels, the sample location will be clearly identified in the field and the adjacent property will be evaluated until the extent of properties containing soil with lead exceedences is defined. A diagram showing the sample locations and where the exceedence occurred will be prepared for each property evaluated.

Utilization of the XRF field-screening device will allow for more expedient decision making regarding the amount of properties with soils containing lead levels requiring removal. The XRF equipment will be calibrated and compared to both known standards and Site specific standards on at least a daily basis in accordance with the standard operating procedure (SOP) for the XRF as presented in Attachment QAPP-F. Site specific standards will be collected and prepared during mobilization activities.

1.4.1.2 Post Excavation Verification Soil Sampling

Post excavation verification samples will be collected from the properties that exhibited soil lead

concentrations in exceedence of the EPA RA levels to verify that the RA objective for soil lead concentrations for residential and industrial/commercial properties of 400 mg/kg and 1,000 mg/kg respectively have been achieved. A grab sample will be collected from the center of each yard or grid of the residential properties depending on the property size and from the center of each grid from the commercial/industrial properties where excavation were soil removal occurred. Samples will be collected from the floor of the excavations and analyzed for total lead.

1.4.1.3 Backfilling

Following soil excavation on the delineated properties, clean imported fill will be used to bring the property back to the original grade. The remediated properties will then be re-vegetated and restored. A ten part composite sample of the backfill material will be collected at the borrow source and analyzed prior to use. Analytical parameters are listed in Table QAPP-1. The frequency and sampling methodology for backfill sources are presented in Table QAPP-2.

1.4.1.4 Waste Characterization

A composite sample covering the entire zone to be excavated will be collected as a five part composite sample from each of the three properties with the highest lead concentrations. The composite sample will be sent to the laboratory and analyzed for Toxicity Characteristic Leaching Procedure (TCLP) lead. If the leachable lead levels exceed the characteristically hazardous level of 5.0 mg/L, the soil excavated from these three properties and all other properties with similar lead levels will be stabilized on-Site prior to placement in the on-Site stockpile or transportation off-Site for disposal. If the TCLP lead results are below 5.0 mg/L, no additional TCLP testing will be required for waste characterization and the soils will be considered non-hazardous for on-Site stockpiling or off-Site disposal.

1.4.1.5 Air Transport Evaluation

An air transport evaluation will be conducted to determine the likelihood of lead impacted soil from the Site re-contaminating the surrounding properties. The air transport evaluation will be conducted by placing sampling apparatus capable of collecting settleable particulate matter at the property boundary at the four compass headings from the Site. The sampling apparatus will consist of laboratory prepared and sealed containers placed at the appropriate locations on-Site and opened to allow particulate matter to settle into them for a period of thirty days. Containers will then be closed and returned to the laboratory so the deposition rate can be reported in grams per square meter for the thirty-day period.

1.4.2 Field Parameters

During the implementation of the RA, the XRF instrument will be used to determine the concentrations of arsenic, lead, cadmium and zinc in soils to delineate the properties requiring soil removal. The XRF will follow calibration procedures using both known and Site specific standards. Acceptable limits of accuracy for the XRF are presented in Section 8, Table QAPP–8.

Other field-monitoring activities will be conducted to collect information regarding worker health and safety in accordance with approved HASP.

1.4.3 Laboratory Parameters

The primary purpose of the RA data collection is to gather sufficient information to determine the extent of metals impacted properties surrounding the Site that exceed the lead action levels for residential and industrial properties of 400 mg/kg and 1,000 mg/kg respectively. The XRF will be used for soil characterization with 10% of the samples also submitted to the approved laboratory to verify the accuracy of the XRF results. In addition, data will be collected to confirm the acceptability of backfill material and to characterize excavated soil for disposal purposes. A summary of the laboratory parameters for the RA and the associated QC samples are provided in Section 3.0, Table QAPP-2.

Acceptable limits on decision errors used to establish the sampling results are provided in Attachment QAPP-C.

1.5 SAMPLE NETWORK DESIGN AND RATIONALE

Total lead analyses will be used as the indicator for contaminant removal in surficial and subsurface soils. Previous sample results from this Site, coupled with experience from similar sites, indicate that not only is lead the predominant contaminant resulting from smelter activities, it is a good general indicator of removal of other metals that may be co-located at the Site.

Table QAPP-2 in Section 3.0 of the QAPP summarizes the project samples to be taken by task, the matrix to be analyzed, the parameters to be analyzed, and the frequency of collection. Project specific reporting limits are presented in Attachment QAPP-B1 through B10.

1.6 PROJECT SCHEDULE

It is estimated that the off-Site investigation and RA will require approximately six weeks to complete. The schedule may be extended based on the number of properties that will require remediation. This will be determined during the off-Site sampling phase.

All efforts will be made to maintain the schedule for completion of the work to be performed as part of the RA as described in the AOC.

TABLE QAPP-1
Intended Data Usage

ACTIVITY	DESCRIPTION	PARAMETERS	INTENDED DATA USAGE
Off-Site Sampling	Soil	All Samples: Arsenic, Lead, Zinc, Cadmium (XRF) 10% of Samples: Total RCRA Metals, Zinc (Laboratory)	Define the extent of properties located near the Site which exceed the residential action level of 400 mg/kg lead or the commercial/industrial action level of 1,000 mg/kg lead
Post Excavation Verification Sampling	Soil	Total Lead	Verify that the RA objectives have been achieved.
Backfill Material Sampling	Soil (Imported Fill)	SVOCs, VOCs, TAL Total Metals, Pesticides/PCBs, Extractable Petroleum Hydrocarbons,	Characterize imported fill material prior to use as backfill on remediated properties.
Waste Characterization Sampling for Stockpiled Soils	Excavated Soils	TCLP Lead	Characterize excavated soils to determine the need for stabilization prior to on-Site stockpiling or off-Site disposal to a Subtitle D Landfill
Air Transport Evaluation	Settleable Dust	Total RCRA Metals	Evaluate the potential for air transport of metals from the Site to adjacent properties

ENTACT
Section
two

2.0 PROJECT ORGANIZATION AND RESPONSIBILITY

2.1 PROJECT ORGANIZATIONAL CHART

The following sections describe the lines of authority of the Project Management Team for overseeing and implementing the required RA at the Site. The ENTACT assigned management team may change during implementation of the RA. If there is a change in the ENTACT management team personnel, the modification will be communicated to the Project Coordinator and the EPA OSC.

2.2 MANAGEMENT RESPONSIBILITIES

EPA On-Scene Coordinator (OSC), Michael Harris

The EPA OSC has the overall responsibility for all phases of the RA.

Project Coordinator, Gary Uphoff, Environmental Management Services Company

The primary responsibility of the Project Coordinator will be to ensure proper coordination among the various project stakeholders. These stakeholders include the EPA, IEPA, the Respondents, and ENTACT.

Project Manager, Rich Wood, ENTACT

The Project Manager will have the overall responsibility for ensuring that the off-Site sampling and RA are implemented and completed in accordance with the AOC, Work Plan, QAPP and federal, state, and local regulations. Specific responsibilities of the Project Manager will include, but are not limited to the following:

- Providing personnel and equipment for remedial activities;
- Ensuring the RA is completed within the approved schedule;
- Ensuring effective communications between the Project Coordinator and the EPA OSC;
- Ensuring that all documents and reports that ENTACT is required to generate meets the requirements of the AOC and Work Plan;
- Communicating any request for modifications to the Work Plan to the Project Coordinator and EPA OSC; and,
- Promptly notifying the Project Coordinator and the EPA OSC in the event unforeseen field conditions and/or problems are encountered.

Field Project Manager, Mr. David Hinton, ENTACT

The Field Project Manager will work with the Project Manager in overseeing the off-Site sampling and RA and ensuring that the activities are implemented and completed in accordance with the AOC, the Work Plan and federal, state, and local regulations. Specific responsibilities of the Field Project Manager will include, but are not limited to the following:

- Providing the Project Manager, the Project Coordinator and the EPA OSC the names and qualifications of the contracted laboratory used to implement the RA;

- Ensuring that ENTACT associates perform their designated duties in accordance with the HASP;
- Ensuring required QA/QC procedures are properly implemented and documented;
- Working with the Project Manager in ensuring the RA is completed following the approved schedule;
- Notifying appropriate personnel identified in the HASP in the event of spills or air releases that exceed criteria;
- Communicating any request for modifications to the Work Plan to the Project Manager, the Project Coordinator and EPA OSC; and,
- Promptly notifying the Project Manager, the Project Coordinator and the EPA OSC in the event any unforeseen field conditions and/or problems are encountered.

Regulatory/Technical Specialist, Ms. Caroline Panico

The Regulatory/Technical Specialist will provide regulatory and technical support to the Project Management Team in ensuring that the Site activities are implemented and completed in accordance with the AOC, the Work Plan and federal, state, and local regulations. She will also provide technical support to the Field Project Manager in the areas of sampling protocol, solid and hazardous waste management, air monitoring, and any other technical design requirements for the RA.

Corporate Health and Safety Officer, Mr. Jonathan Patlak, ENTACT

The Corporate Health and Safety Officer will coordinate and provide oversight for the health and safety issues at the Site. He will be responsible for preparing the HASP and conducting the health and safety orientation meeting prior to implementing the RA. He will review weekly health and safety updates from the Site and conduct several inspections at the Site throughout the duration of the RA.

Management Control Process

The ENTACT Project Manager has overall responsibility for successfully completing the RA. This includes safely completing AOC and Work Plan items, fulfilling contractual obligations, compliance with the QAPP and HASP, and meeting or exceeding the established project schedule and budget. The Project Manager will accomplish these objectives by monitoring the work progress, reviewing and planning each project task with experienced technical staff and the Field Project Manager, and ensuring that appropriate and sufficient resources are available to the Field Project Manager and the On-Site QA/QC Officer.

The Project Manager will receive daily progress reports from Site personnel appraising him of the status of planned, ongoing, and completed work, including QA/QC performance and health and safety issues. In addition, the Project Manager will be notified of any potential problems and recommendations for solutions and/or corrective action.

Qualifications and experience of ENTACT's Management Team are provided in Attachment QAPP-A of the QAPP.

2.3 QUALITY ASSURANCE RESPONSIBILITIES

EPA Region 5 Superfund Quality Assurance Coordinator

The EPA Superfund Quality Assurance Reviewer has the responsibility to review and approve the QAPP. In addition, the EPA Quality Assurance Coordinator is responsible for conducting external performance and system audits of the laboratory and evaluating analytical field and laboratory procedures.

Quality Assurance (QA) Manager, Caroline Panico, ENTACT

The ENTACT QA Manager will be responsible for ensuring that all required procedures for this project are being followed. In addition, the ENTACT QA Manager will be responsible for the data validation of all sample results from the analytical laboratory. Specific responsibilities will include, but are not limited to the following activities:

- Provide required QC testing is performed and documented and the results are provided to the ENTACT's project management team, the Project Manager, and EPA in accordance with the requirements of the Work Plan and QAPP;
- Provide oversight and direction to the on-Site Quality Assurance/Quality Control Officer; and,
- Provide assistance in the modification of QA methodology or implementation based on conditions encountered during the remedial activities if different than specified in the approved QAPP.

On-Site Quality Assurance/Quality Control (QA/QC) Officer, ENTACT and CEC

The On-Site QA/QC Officer will be responsible for performing required QC testing at the Site. The QA/QC Officer will operate independently of the ENTACT Project Manager and Field Project Manager. The QA/QC Officer will communicate any QA/QC issues related to the Site to the QA Manager. The QA/QC Officer will have the authority to correct and implement additional measures to assure compliance with the AOC, Work Plan and the QAPP. Specific responsibilities will include, but are not limited to the following activities:

- Document any deviations to the Work Plan with a justification for the deviations, and if necessary appropriate notification;
- Secure necessary sampling tools, bottles, packaging/shipping supplies, chain-of custody (COC) documents, etc.;
- Collect or direct the collection of samples and ship samples at the required frequencies and for laboratory analysis parameters specified in the QAPP;
- Document the location, time, and date of all samples that are collected and shipped to the laboratory;
- Document the location, time, and date of all samples that are collected and analyzed in the field;
- Interface with the sampling technicians to ensure the sample collection is coordinated with the general progression of the work;
- Notify the Project Manager, Project Coordinator and the EPA of any sampling activities associated with the implementation of the AOC and Work Plan; and,
- Obtain analytical results and report the data to the Project Manager, Project Coordinator, and the EPA OSC.

2.4 LABORATORY RESPONSIBILITIES

The laboratory that will be performing the soil and air sample analysis for this project is:

Severn Trent Laboratories (STL)

13715 Rider Trail North
Earth City, Missouri 63045
Phone (314) 298-8566

STL Project Manager, John Powell

The STL Project Manager will report directly to the ENTACT QA Manager and will be responsible for ensuring that all resources of the laboratory are available as required. He is also responsible for the overview of the final analytical reports.

STL Quality Assurance Officer

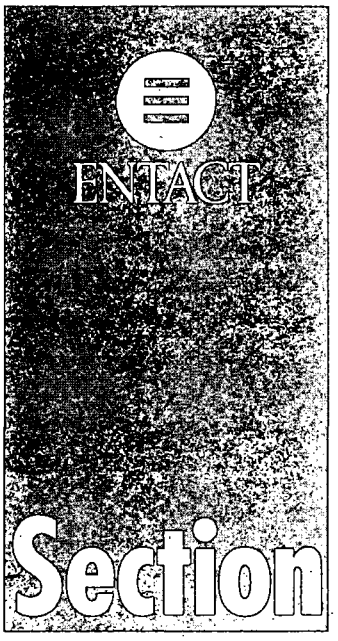
The STL QA Officer has the overall responsibility for data generated by the laboratory. The STL QA Officer will communicate data issues through the STL Project Manager. In addition, the STL QA Officer will overview laboratory quality assurance and QA documentation, conduct detailed data review, determine whether to implement corrective action, and define appropriate laboratory procedures.

STL Sample Custodian

The STL Sample Custodian will report to the STL Project Manager. The responsibilities of the STL Sample Custodian will include: receiving, recording and inspecting the incoming samples; verifying the COC and checking for accuracy; notifying the laboratory manager and supervisor of sample receipt and inspection; assigning a unique identification number and customer number, and entering each number into the sample receiving log; and transferring samples to the appropriate laboratory section.

STL Technical Staff

The STL Technical Staff will be responsible for sample analysis and identification of corrective actions.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Section
three

3.0 QUALITY ASSURANCE OBJECTIVES FOR MEASUREMENT DATA

The overall QA objective for this project is to develop and implement procedures for field sampling, COC, laboratory analysis, and reporting that will provide results that are legally defensible in a court of law. The purpose of implementing these procedures is to assess the data generated for PARCC for both the laboratory analytical program and field sample collection activities. The primary goal of the program is to ensure that the data generated are representative of environmental conditions at the Site. To obtain this goal, a combination of statistical procedures and qualitative evaluations will be used to check the quality of the data.

PARCC will be computed in the manner described in the following paragraphs. A qualitative assessment of PARCC factors will be made and will be documented. Specific procedures for sampling, COC, laboratory instrument calibration, laboratory analysis, reporting of data, internal QC, audits, preventative maintenance of field equipment, and corrective action are described in other sections of this QAPP.

3.1 PRECISION

The precision of laboratory results and field sampling efforts will be evaluated by examining laboratory and field QC sample results. Analytical precision will be evaluated for analytical methods by comparing the QC criteria stipulated in the SOPs to the results from laboratory matrix spike (MS)/matrix spike duplicate (MSD) samples and field duplicate samples.

3.1.1 Definition

Precision is a measure of mutual agreement among individual measurements of the same property, usually under prescribed similar conditions, usually expressed in terms of the standard deviation.

3.1.2 Field Precision Objectives

Field precision is assessed through the collection and measurement of field duplicates at a rate of 1 duplicate per 10 investigative analytical samples submitted to the laboratory.

3.1.3 Laboratory Precision Objectives

Precision in the laboratory is assessed through the calculation of relative percent differences (RPD) for replicate samples. The equations to be used for precision in this project can be found in Section 12 of this QAPP. Precision control limits are provided in Attachment QAPP-C.

3.2 ACCURACY

By examining the results obtained from the analysis of duplicate samples, laboratory MS/MSD samples, and equipment field blank samples, the accuracy of the analytical data will be assessed. One duplicate sample will be collected for every 10 investigative samples. One MS, and one MSD will be analyzed for every 20 investigative samples. One equipment field blank sample will be prepared for every 10 investigative samples. Field blanks will only be collected if disposable-sampling equipment is used to verify that decontamination procedures are adequate and not biasing data.

Data will be qualified in accordance with the appropriate EPA functional guidelines for evaluating data if either field QC blanks or laboratory QC blanks indicate that the accuracy or precision of analytical results is compromised.

3.2.1 Definition

Accuracy is the degree of agreement of a measurement with an accepted reference or true value.

3.2.2 Field Accuracy Objectives

Accuracy in the field is assessed through the use of field blanks and adherence to all sample handling, preservation, and holding times.

3.2.3 Laboratory Accuracy Objectives

Laboratory accuracy is assessed through the analysis of MS or standard reference materials (SRM) and the determination of percent recoveries. The equation to be used for accuracy in this project can be found in Section 12 of this QAPP. Accuracy control limits are provided in Attachment QAPP-C of the QAPP.

3.3 COMPLETENESS

3.3.1 Definition

Completeness is the amount of valid data obtained from a measurement system compared to the amount that was expected and required to meet the project data goals.

3.3.2 Field Completeness Objectives

Field completeness is the measurement of the amount of valid measurements obtained from all the measurements taken during the project. The intent of this program is to attempt to achieve a goal of 100 percent completeness. Realizing that under normal conditions this goal may not be achievable, the completeness goal for this program is 90 percent. This completeness goal is considered adequate to meet the data quality objectives for this Site based on prior consideration of PARCC parameters, the sampling design plans, and data collection activities proposed for each medium. In developing the sampling design plan, critical data points were carefully considered and identified to help ensure comparability of data. The equation for completeness is presented in Section 12 of this QAPP.

3.3.3 Laboratory Completeness Objectives

Laboratory completeness is a measure of the amount of valid measurements obtained from all the measurements taken during the project. The intent of this program is to attempt to achieve a goal of 100 percent completeness. Realizing that under normal conditions this goal may not be achievable, the completeness goal for this program is 90 percent. The laboratory equation for completeness is presented in Section 12 of this QAPP.

3.4 REPRESENTATIVENESS

Representativeness expresses the degree to which sample data accurately and precisely represent environmental conditions and parameter variations at a sampling location. Representativeness is a qualitative parameter most concerned with the proper design of the sampling program. Assuring that sampling locations are properly selected and a sufficient number of investigative samples are collected best satisfies the representativeness criterion.

3.4.1 Definition

Representativeness is the selection of analytical methods and sampling protocols and locations such that results are representative of the media being sampled and conditions being measured.

3.4.2 Measures to Ensure Representativeness of Field Data

Representativeness is dependent upon the proper design of the sampling program and will be satisfied by ensuring that proper sampling techniques are used.

3.4.3 Measures to Ensure Representativeness of Laboratory Data

Representativeness in the laboratory is ensured by using the proper analytical procedures, meeting sample-holding times, and analyzing and assessing field duplicate samples. The sampling network was designed to provide data representative of facility conditions. During the development of this network, consideration was given to past Site activities, physical setting, and constraints inherent to the Work Plan. The rationale of the sampling network is discussed in detail in Section 4 of this QAPP.

3.5 COMPARABILITY

Comparability cannot be ensured through use of standard methods and protocols alone. In order to compare data, various important elements will be considered. During this project, three elements will be evaluated for data comparability. These three elements include analytical methods, quality of data, and sampling design. If after the initial evaluation, data do not appear comparable, the QA Manager will attempt to identify other components possibly affecting comparability, including but not limited to field conditions, sampling protocols, and the occurrence of true data anomalies.

3.5.1 Definition

Comparability is an expression of the confidence with which one data set can be compared to another.

3.5.2 Measures to Ensure Comparability of Field Data

Comparability is dependent upon the proper design of the sampling program and will be satisfied by ensuring that proper sampling techniques are used.

3.5.3 Measures to Ensure Comparability of Laboratory Data

Planned analytical data will be comparable when similar sampling and analytical methods are used and documented. Similar QA objectives will be used throughout the project to ensure comparability.

3.6 LEVEL OF QUALITY CONTROL EFFORT

Field equipment blank, duplicate, and MS samples will be analyzed to assess the quality of data resulting from the field sampling and analytical programs.

3.6.1 Field Data

One field equipment blank will be prepared for every 10 investigative samples if reusable-sampling equipment is used. Sampling procedures are specified in Section 4 of this QAPP.

The precision and accuracy of field measurements are discussed in Section 8.1 of the QAPP and listed in Table QAPP-8.

3.6.2 Laboratory Data

Method blank samples are generated within the laboratory and used to assess contamination resulting from laboratory procedures. Field duplicate samples are analyzed to check for sampling and analytical reproducibility. Matrix spikes provide information about the effect of the sample matrix on the digestion and measurement methodology. All MS are performed in duplicate and are hereinafter referred to as MS/MSD samples. One MS/MSD will be analyzed for every 20 or fewer investigative samples per sample matrix.

Table QAPP-2
SUMMARY TABLE OF SAMPLING AND ANALYSIS PROGRAM FOR THE REMOVAL ACTION
OLD AMERICAN ZINC SITE

Sample Type	Analysis Parameters	Investigative Samples			Field Quality Control Samples									Totals
					Field Duplicates			Field Blanks[4]			MS/MSD[1]			
		No.	Freq.	Total	No.	Freq.	Total	No.	Freq.	Total	No	Freq.	Total	Total
Off-Site Sampling	Laboratory ---- RCRA Metals, Zinc[6]	50+	1	50+	5	1	5	5	1	5	--	--	--	60
	Field ---- XRF Arsenic, Lead, Zinc, Cadmium	500+	1	500+	--	--	--	--	--	--	--	--	--	500
Verification Sampling	Laboratory----Total Lead	500+	1	500+	50	1	50	50	1	50	--	--	--	600
Backfill Testing[3]	Laboratory --- RCRA Metals[2]	2	1	2	--	--	--	--	--	--	--	--	--	2
	Laboratory --- VOCs	2	1	2	--	--	--	--	--	--	--	--	--	2
	Laboratory --- SVOCs	2	1	2	--	--	--	--	--	--	--	--	--	2
	Laboratory --- Pesticides/PCBs	2	1	2	--	--	--	--	--	--	--	--	--	2
	Laboratory---Extractable Petroleum Hydrocarbon	2	1	2	--	--	--	--	--	--	--	--	--	2
	Laboratory---TAL Total Metals	2	1	2	--	--	--	--	--	--	--	--	--	2
Waste Characterization Sampling[5]	Laboratory ---- TCLP Lead	1+	1	1+	--	--	--	--	--	--	--	--	--	1+
Air Transport Monitoring	Laboratory----RCRA Metals	4	1	4	--	--	--	--	--	--	--	--	--	4

NOTES:

[1] For metals analysis, no extra sample volume is required; MS/MSD will be performed at a rate of one per twenty investigative samples analyzed by the laboratory.
[2] RCRA Metals = arsenic, barium, cadmium, chromium, lead, selenium, silver, and mercury.
[3] Estimate of one sample to be collected for every 5,000 yards of material per source. If more than one source is used, additional sampling will be required.
[4] Field blank samples are only required if re-usable sampling equipment is used (i.e. stainless steel bowls or trowels).
[5] Assumes analysis of one composite sample collected from the three properties with highest concentration of lead
[6]Total digestion with hydrofluoric acid utilizing methods consistent with the EPA Contract Laboratory Program

ENTACT
Section
four

4.0 SAMPLING PROCEDURES

This section summarizes the sample documentation, sampling procedures and the QC sample preparation requirements associated with the RA tasks.

4.1 SAMPLE DOCUMENTATION/IDENTIFICATION

The sampling activities and examples of the identification coding system associated with each type are listed below with an explanation:

Samples Type	Identification System
Air Samples:	
Settleable Particulate Matter	SPM-Unit#-000
Personal/Area Low Volume Samples	PAS-Unit#-000
Soil Samples:	
X-Ray Fluorescence Field Analysis: Residential Properties (<10,000 ft^2) Residential Properties (>10,000 ft^2) Commercial/Industrial Properties	 X-000R-BY-6 X-000R-BY1-6 X-000C-1-6
Confirmatory Laboratory Samples: Residential Properties (<10,000 ft^2) Residential Properties (>10,000 ft^2) Commercial/Industrial Properties	 L-000R-BY-6 L-000R-BY1-6 L-000C-1-6
Verification Sampling: Residential Properties Commercial/Industrial Properties	 V-000R-BY-6 V-000C-1-6
Imported Backfill Samples	BF-000
Treated Soil Samples (stabilized soil, if needed)	TF-000
Waste Characterization Samples:	
Solid Waste (stabilized soils, if needed)	WC-000
Quality Control Samples:	
Field Duplicate Samples for Soil	L-000-FD
Field Equipment Blanks (if disposable equipment is used)	FB-000

In general, all numbering sequences shown above with "000" will begin with the number "001" and will continue upward by one unit (i.e., X-001, X-002, X-003, etc.) until the final sample for that sample type has been collected.

Off-Site soil samples analyzed in the field with the XRF equipment will be numbered for incorporation into the XRF database. The samples will be numbered using the property identification number, the specific sample location within the property and the depth at which the sample was collected.

Properties will be numbered sequentially with the property closest to the Site numbered 001 and continuing upward as sampling progresses away from the Site. For residential properties, the letter "R" will follow the property number. For commercial/industrial properties the letter "C" will follow the property number.

For residential properties less than 10,000 square feet in size, a composite sample will be collected from the front yard (FY) and back yard (BY) at the three specified depth intervals of 0-6 inch, 6-12 inch, and 12-18 inch. A composite sample collected at a depth of 6 inches from the back yard of residential property 001 will be numbered X-001R-BY- 6.

For residential properties greater than 10,000 square feet in size, the yard will be divided into four equal size grids. A composite sample will be collected from each of the four grids in the yard at the three specified depths. The sample numbers for these larger properties will include the grid number (1-4) after the sample location. A composite sample collected at a depth of 6 inches from grid #4 in the back yard of residential property 001 will be numbered X-001R-BY4- 6.

For all residential properties, a composite sample will be collected from the drip zone (DZ) around the perimeter of the residences from the 0-6 inch depth. A sample collected from the drip zone of residential property 001 will be numbered X-001R-DZ. Because all drip zone samples will be collected from the same depth interval of 0-6 inch, the sample depth will not be included in the sample identification number.

For commercial/industrial properties less than 20,000 square feet in size, the property will be divided into two equal size grids. A composite sample will be collected from each of the two grids at the three specified depths. For industrial properties greater than 20,000 square feet in size, the property will be divided into four equal size grids. A composite sample will be collected from each of the four grids at the three specified depths. The sample numbers for the industrial properties will include the property identification number, the sample location grid number, and the depth at which the sample was collected. A composite sample collected at a depth of 6 inches from grid #4 on industrial property 001 will be numbered X-001C-4- 6.

After field analysis of the samples utilizing the XRF is complete, 10% of the XRF samples collected will be sent to the laboratory to confirm the XRF field results. Laboratory confirmation samples will be identified using the same method as the XRF samples but the "X" in the sample ID number will be replaced with an "L".

For properties that require soil removal, post excavation verification samples will be collected and analyzed for total lead to verify that the RA objectives for residential and commercial/industrial properties

have been met. Verification samples will be identified using the same method as the XRF samples but the "X" in the sample ID will be replaced with the letter "V".

Sample identification documents will be carefully prepared to maintain identification and COC records, and to control sample disposition. Components of the field documentation procedures include the use of field logbooks, sample labels, and the COC forms. Original data recorded in field logbooks, COC records, and other forms will be written in waterproof ink. None of these documents will be altered, destroyed, or discarded, even if they are illegible or contain inaccuracies that require a replacement document. If an error is made on a document assigned to one individual, that individual will make the corrections by making a line through the error, entering the correct information, and initialing and dating the change. Samples and documentation will be maintained and handled by as few people as possible.

4.2 SAMPLE COLLECTION/PREPARATION PROCEDURES

4.2.1 XRF Field Analysis

The XRF instrument will be utilized to allow field analysis during the off-Site-sampling event to define the extent of properties surrounding the Site that have soil lead concentrations in exceedence of the EPA RA levels. Utilization of the XRF field-screening device will allow for more expedient decision making regarding the amount of properties with soils containing lead levels requiring removal. Ten percent of the XRF samples will be confirmed by laboratory analysis.

XRF field analysis of the residential properties less than 10,000 square feet will consist of a composite sample collected from the front yard and from the back of the house at the three specified depths of 0-6 inch, 6-12 inch and 12-18 inch. Each composite sample will consist of five sample aliquots with one aliquot collected at each of the four corners of the area to be sampled and one collected near the center. A total of three composite samples will be collected in each area to be sampled consisting of one composite sample for each of the three specified depths.

For residential properties greater than 10,000 square feet in size the total yard space will be divided into four equal size grids. A composite sample will be collected from each of the four grids at the three specified depths of 0-6 inch, 6-12 inch and 12-18 inch. Each composite sample will consist of five sample aliquots with one collected at each of the four corners of the grid to be sampled and one collected near the center. A total of three composite samples will be collected in each grid to be sampled consisting of one composite sample for each of the three specified depths.

A composite sample representing the drip zone will also be collected around the perimeter of the residence on all residential properties. The drip zone composite sample will consist of one grab sample collected on each of the four sides of the residence at the 0-6 inch depth interval.

Commercial/industrial properties will be sampled using the same protocol as specified for the residential properties. Industrial properties less than 20,000 square feet in size will be divided into two equal size grids. A 5-part composite sample will be collected from each of the two grids at the three specified depths. For industrial properties greater than 20,000 square feet in size, the property will be divided into four equal size grids. A 5-part composite sample will be collected from each of the four grids at the three specified depths.

The sample collection frequency for the residential and commercial/industrial properties will be decreased once the XRF results consistently indicate that the impacted soils are limited to the upper intervals. After concurrence with the EPA OSC, sampling depths will be limited to the 0-6 inch interval.

The aliquot samples will be collected as a grab sample using the following equipment and supplies:

- Stainless steel auger
- Stainless steel or plastic disposable scoops or trowels
- Stainless steel bowl
- Sample containers and plastic bags
- Field notebook
- Decontamination supplies (Decontamination may be conducted at the sample location staging area or the main decontamination area)

Soil samples will be collected using either a stainless steel soil auger or a trowel. The auger will be capable of retrieving soil samples at the specified 6-inch intervals and will have a turn bar that can be twisted to advance the probe to the desired depth up to 18 inches. If a trowel is used, measurements of the sample depth will be made manually as the sampling depth progresses. Each of the five aliquot samples for each depth will be placed in a Ziploc bag and properly labeled with the sample identification number, sample depth, time, date and initials of sampler. Field notes will be recorded for each sample taken and will include soil description (color, type, and foreign material) and any other pertinent observations relating to the sample or Site conditions at the time of sampling. The bagged samples will be transported to the on-Site soil sample and managing area stationed in a mobile trailer. The five aliquot samples for each sample location and sample depth will be transferred to a stainless steel bowl and thoroughly mixed to achieve a homogenous blend.

Preparation of the composite samples collected from the 0-6 inch depth interval will be performed in accordance with the TRW Recommendations for Sampling and Analysis of Soil at Lead (Pb) Sites (OSWER 9285.7-38, dated April 2000). According to this guidance document, "the preference is for analysis of the fine fraction only, because it provides the best characterization of the current risk from exposure by incidental ingestion." After homogenizing the composite samples collected from 0-6 inch, the sample will be placed in a microwave and dried. The dried sample will then be placed in a series of sieves to remove the coarse fraction. The fine fraction is the portion of the sample that passes through a 250-micron (No. 60) sieve (ASTM 1999). XRF and laboratory analysis of all soil samples collected at the surface of the residential and industrial properties (0 to 6-inch depth) will be performed on the fine fractions.

All samples will be analyzed for arsenic, lead, zinc, and cadmium using the XRF equipment. To ensure a controlled environment, analysis will be performed in the on-Site soil sample and managing area stationed in a mobile trailer. The XRF equipment will be calibrated and compared to known standards and Site specific standards on a routine basis in accordance with the SOP for the XRF. The particular instrument to be used is the Spectrace 9000 Portable XRF Analyzer. This device utilizes a probe, which consists of a sealed aluminum enclosure containing a high-resolution mercuric iodide detector and three radioisotope x-ray excitation sources, Fe-55, Cd-109 and Am-241. The Spectrace 9000 utilizes a fundamental parameter XRF calibration derived from theoretical considerations. The menu-driven software supports multiple XRF calibrations called "Applications." Each Application is a complete analysis configuration including elements to be measured, interfering elements in the sample, and a set of

fundamental parameter calibration coefficients. The SOP for the XRF instrument is included in Attachment QAPP-F.

4.2.2 Laboratory Confirmation Sampling

To provide confirmation of the XRF analysis conducted in the field, ten percent of the samples analyzed with the XRF will be submitted to the laboratory for total digestion with hydrofluoric acid and analysis of the RCRA metals and zinc utilizing methods consistent with the EPA Contract Laboratory Program. Laboratory confirmation samples will be prepared by obtaining an appropriate volume of sample from the samples that were previously prepared for XRF analysis. The laboratory confirmation samples will be selected to be representative of the range of lead concentrations measured by the XRF.

The selected sample will be placed in an appropriate sample jar and a new sample identification number will be assigned to indicate it is a laboratory sample (i.e. the "X" designation will be replaced with an "L").

Laboratory sample results will be correlated to the XRF field analysis sample results to confirm the accuracy of the XRF results obtained in the field. Laboratory data results will be utilized to determine the correlation of XRF on-Site laboratory results to meet the RA objectives. The average percent difference between the XRF data and the laboratory data will be calculated.

4.2.3 Post-Excavation Verification Sampling

To verify that the RA objectives have been achieved, post excavation verification samples will be collected from the center of the yards or grids of the residential and commercial/industrial properties that required soil removal. One grab samples will be collected from the floor of the excavation and placed in a Ziploc bag using the same sampling protocol as described for the XRF field analysis sample collection. The bagged grab sample will be transported to the on-Site soil sample and managing area stationed in a mobile trailer.

The bagged verification sample will be placed in an appropriate sample jar and a sample identification number will be assigned to indicate it is a post excavation verification sample. Verification samples will be sent to the approved laboratory and analyzed for total lead.

4.2.4 Backfill Characterization Sampling

Backfill samples will consist of a ten part composite sample collected at each borrow source location for each 5,000 cubic yards of imported material prior to shipment to the Site to ensure the material meets the project requirements. An area representing 5,000 cubic yards will be divided into ten equal sized areas of in place material and an aliquot sample will be collected from each area. Field notes will be recorded for each sample taken and will include soil description (color, type, and foreign material) and any other pertinent observations relating to the sample or Site conditions at the time of sampling.

With the exception of the volatile organic compound (VOC) samples, each of the ten aliquots will be collected using a trowel and placed in either a clean Ziploc bag or stainless steel bowl and thoroughly mixed to achieve a homogeneous blend. The composited soils will then be placed directly into the appropriate clean jars supplied by the laboratory. VOC samples will be collected by placing the sample

aliquot directly into the sample jar and covering the sample tightly between each aliquot collection until all ten aliquots are collected. The VOC sample jar will be packed tightly after collection of the last sample to eliminate headspace. This will be done to minimize any loss of volatiles through manual disturbance of the soils.

The jar will be properly labeled with the sample identification number, sample depth, time, date and initials of sampler. The samples will be placed in an iced cooler and submitted to the off-Site laboratory. No field duplicates, field blanks or MS/MSD samples will be collected for the backfill samples.

The samples will be submitted to the designated Project Laboratory, STL in St. Louis Missouri, for chemical analysis of the applicable parameters using DQO Screening Level 3 in accordance with the QAPP. DQO Screening Level 3 will provide the appropriate level of quality assurance data for backfill material characterization.

4.2.5 Waste Characterization Sampling

To characterize the removed soils, one composite sample will be collected from each of the three properties with the highest recorded soil lead concentrations. For each of the three properties identified, a five part composite sample will be collected in the specific sample location with the highest lead concentration.

The composite samples will consist of five sample aliquots with one aliquot collected at each of the four corners of the area to be sampled and one collected near the center. Each of the five aliquots will be placed in a Ziploc bag and properly labeled with the sample identification number, sample depth, time, date and initials of sampler. The bagged samples will be transported to the on-Site soil sample and managing area stationed in a mobile trailer. The five aliquot samples will be transferred to a stainless steel bowl and thoroughly mixed to achieve a homogenous blend. The samples will be transferred to an appropriate sample container and prepared for submittal to the laboratory.

The samples will be submitted to the designated Project Laboratory, STL in St. Louis Missouri, for analysis of TCLP in accordance with the QAPP. DQO Screening Level 3 will provide the appropriate level of quality assurance data for waste characterization.

4.2.6 Air Deposition Sampling

Air deposition sampling will be performed as part of an air transport evaluation to determine the likelihood of lead impacted soil from the Site re-contaminating the surrounding properties. Sample collection and analysis for the air transport evaluation will be conducted in accordance with ASTM D 1739-98, Standard Test Method for Collection and Measurement of Dustfall (Settleable Particulate Matter)

Sampling apparatus capable of collecting settleable particulate matter will be placed along the property boundary of the Site at the four compass headings. Sampling apparatus will consist of stainless steel or weatherproof plastic containers with tight fitting lids. The containers and lids will be thoroughly cleaned with a detergent solution and properly labeled with the date, time, and sample identification. The sample container will be placed on a stand tall enough to allow the top of the container to be at a height of 2 meters above the ground and the container lids will be removed. The sampling apparatus will remain in

place for a period of thirty days. At the end of the sampling period, the containers will be removed from the stands, the lids will be placed on the container and the date and time will recorded.

The sampling apparatus will submitted to an off-Site laboratory where the volume of particulate matter collected in the container will be determined and the deposition rate will be calculated in grams per square meter.

The samples will be submitted to the designated Project Laboratory, STL in St. Louis Missouri, for analysis of TCLP in accordance with the QAPP.

4.3 FIELD QC PROCEDURES

Field duplicate samples will be collected for laboratory confirmation soil samples at a rate of one duplicate for every ten composite samples collected. At the designated sample location where a duplicate sample will be collected, an ample volume of material will be placed in a Ziploc plastic bag while collecting the grab samples and thoroughly homogenized prior to filling the sample jars. The field duplicate sample will be blind labeled as L-001FD and continue sequentially from 001 with the associated laboratory confirmation sample recorded in the logbook.

If reusable-sampling equipment is used, (i.e. stainless steel bowl and/or trowel), a field blank sample will be prepared at a rate of one rinsate sample for every 10 investigative samples taken by pouring distilled water over the decontaminated sampling equipment.

MS/MSD samples will be performed at a rate of one for every 20 investigative samples analyzed by the laboratory. No extra sample volume is required for the MS/MSD samples for metals. The MS/MSD will be performed at a rate of one per twenty investigative samples.

The samples will be submitted to the designated Project Laboratory, STL in St. Louis Missouri, for analysis of TCLP in accordance with the QAPP.

4.4 SAMPLE CONTAINERS, PRESERVATIVES AND VOLUME REQUIREMENTS

Laboratory confirmation samples will be placed into clean plastic or glass 2- or 4-ounce containers for soil samples and 8-ounce containers for TCLP lead analysis. Sample jars will be supplied by a vendor or laboratory and will be certified clean. There are no preservatives required for either analyses and the container should be completely filled. The container will be labeled with the sample identification number, date and time of sampling and the initials of the sampler. The sample container will be placed in a sealed plastic bag for transportation to the laboratory. The designated laboratory may provide a daily courier service during removal activities to allow for an expedited analytical turn-around time. If samples must be transported by means of commercial transportation, the samples will be placed in a cooler, packaged in a manner to prevent shifting and breakage in transit, and a custody seal will be placed on the cooler housing the samples such that any tampering with the cooler will be evident by the seal. No ice is required for metal parameters. Sample labels and custody seals are presented in Attachment QAPP-D.

Backfill samples that include multiple parameters will be placed into the appropriate container. The VOC sample will be collected first and placed directly into the sample container to minimize any loss of volatile compounds, with no mixing or homogenizing the soils to prevent loss of potential volatile

contaminants. Samples to be analyzed for multiple parameters will be placed on ice.

Sample containers and preservatives are not required for the XRF screening samples. Clean zip-lock bags will be used as sample containers. These bags will be labeled to identify the sample identification number, date, time, and sampler's initials.

Air sample containers will not be open, left out or tampered with before or after the thirty-day sampling period. Containers will be tightly sealed at the end of the sampling period. There are no preservatives required for lead for settleable dust analysis.

4.5 SAMPLE CUSTODY

A COC form will be filled out at the time of sampling. Information to be recorded on the COC includes sample identification, sample description, name(s) of sampler(s), and requested analyses. The COC will be placed in a sealed plastic bag for protection and will accompany the associated samples to the laboratory. Any time the sample custodian changes, the person relinquishing the samples shall sign the COC and note the date and time of transfer. The person receiving the samples shall also sign the COC and note the date and time of transfer. Attachment QAPP-D of the QAPP includes examples of COC forms to be used.

4.6 DECONTAMINATION PROCEDURES

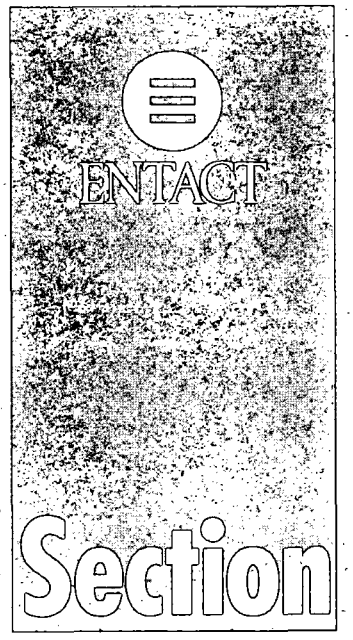
All re-usable sampling equipment will be decontaminated utilizing a triple rinse procedure. During this procedure, the sampling equipment is scrubbed in a potable water/detergent wash (gross rinse), rinsed in potable water (intermediate rinse), and rinsed with distilled water (final rinse). All three decontamination fluids are changed as needed to ensure proper decontamination; however, to conserve the quantity of waste generated, ENTACT will downgrade the three phase fluids. For example, the final phase fluids are downgraded to intermediate fluids, intermediate fluids are downgraded to gross fluids, gross fluids are collected in a DOT approved container, and fresh distilled water is placed in the final phase. This method minimizes waste and ensures that the final phase fluids are clean. Spent decontamination fluids will be collected throughout the project for proper disposal at an authorized treatment facility.

After decontamination, the sampling equipment will be dried with disposable towels and stored in plastic sampling tool boxes between sampling events. All decontaminated equipment within the sampling tool box will be placed in individual plastic bags or wrapped in disposable towels. The sampling tool boxes will also be decontaminated weekly to ensure cleanliness. All trash and personal protective equipment (PPE) generated during sampling will be placed in designated disposal containers for such items.

4.7 SAMPLE PACKAGING AND SHIPMENT PROCEDURES

Sample containers will be laboratory prepared and shipped in sealed containers to assure that they remain clean. Sample containers will be selected to ensure compatibility with the media being collected, preserve sample integrity, and minimize breakage during transportation. Sample labels will be filled out at the time of sampling and will be affixed to each container to identify sample number, sampler's name, date and time of collection, location of sampling point, and project identification data.

After the containers for a given sampling location have been filled out, they will be placed in plastic Ziploc storage bags, placed on ice (for VOC, Semi Volatile Organic Compounds (SVOC) and pesticide/polychlorinated biphenyls (PCB) samples only), in an insulated cooler, to be delivered to STL. Each sample container will be secured in packing material, as appropriate, for shipment. The insulated cooler lid will be taped closed and sealed to avoid the entrance of contaminants into the cooler and to avoid leaking from the cooler. Shipment of samples to the laboratory will take place on the same day as collection. The COC form will be enclosed in a sealed plastic bag and adhered inside the sealed cooler. If the samples are sent by common carrier, a bill of lading will be used to document the custody of the sample while in transit. Commercial carriers are not required to sign the COC forms as long as the forms are sealed inside the cooler.


ENTACT
Section
five

5.0 CUSTODY PROCEDURES

Custody is one of several factors necessary for the admissibility of environmental data as evidence in a court of law. Custody procedures help to satisfy the two major requirements for admissibility: relevance and authenticity. Sample custody is addressed in three parts: field sample collection, laboratory analysis, and final evidence files. Final evidence files, including all original laboratory reports, are maintained under document control in a secure area.

A sample or evidence file is under one's custody if:

- the item is in actual possession of a person; or
- the item is in the view of the person after being in actual possession of the person; or
- the item was in actual physical possession but is locked up to prevent tampering; or
- the item is in a designated and identified secure area.

5.1 FIELD CUSTODY PROCEDURES

Sample identification documents will be carefully prepared to maintain identification and COC records and to control sample disposition. Components of the field documentation procedures include the use of field logbooks, sample labels, and the COC forms. Original data recorded in field logbooks, COC records, and other forms will be written in waterproof ink. The field sampler is personally responsible for the care and custody of the samples until they are transferred or properly dispatched.

5.1.1 Field Logbook Records

A field log of daily activities will be used to record sampling activities on a daily basis. This book will be bound and have consecutively numbered pages. Entries in the field logbook will be made in ink and will include: the name of the author; date and time of entry; location of activity; names and affiliations of personnel on-Site; sample collection or measurement methods; number of samples collected; daily weather report; sample identification numbers; field observation and comments; sampling depth increment for soils; field measurements; locations of photographs; and any deviations from the sampling plan. Each logbook will be assigned a project specific document number. The field logbook will be stored in the job trailer when it is not in use.

5.1.2 Sample Labels

Sample labels are necessary to prevent misidentification of samples. Preprinted labels will be provided prior to the sampling activities. Each label will contain space for the following information: name of Site, sample identification, date and time of sample collection, media sampled, name of sampler, and types of analyses to be performed. Example of custody seal and label is provided in Attachment QAPP-D of the QAPP.

5.1.3 Chain-of-Custody Records

A COC form will be completed to record the custody of every sample collected for laboratory submission. A COC form will accompany every shipment of samples to the analytical laboratory in order

to establish the documentation necessary to trace sample possession from the time of sample collection through sample analysis.

The sample portion of the COC form will include the following:

- Project number, name and location;
- Sample identification;
- Name of Project Manager, Sampler, and Recorder;
- Sampling information (sampling area, depth, media type, type of sample, date and time of collection, etc.);
- Analysis to be performed;
- Preservatives used, if any; and
- Signatures of persons involved in the COC possession, including dates.

When a COC form is filled out, one page of the three-part form is retained and placed in a file at the on-Site office. The other two parts of the form accompany the sample to the laboratory. The laboratory retains one of the two pages and the other is returned with the sample result report. When the sample report is received, it is cross-checked with the COC file record and both COC pages and the laboratory report are placed in a file in fireproof storage at the on-Site office. The analytical result is also entered into a computer database consisting of a comprehensive list of all samples taken at the Site and the analytical results.

5.2 LABORATORY CUSTODY PROCEDURES

Samples, which are delivered by clients or received by courier, are placed in a secure Sample Control Area immediately upon delivery. Coolers containing samples are unpacked within ½ hour of receipt or placed in the walk-in cooler until unpacked. The COC accompanying the samples will be signed by the Sample Custodian or their designee at the time of delivery by the client, or in the case of courier delivery, where the COC is sealed up inside of the cooler, at the time of unpacking.

At the time of arrival and/or unpacking, coolers will be inspected for evidence of damage. They will be unpacked carefully and samples will be organized on the lab bench in numerical order or by sample sets and assigned a laboratory job number. The condition of both shipping containers and sample containers will be recorded on the internal COC form.

Information on the COC shipped with samples will be verified and recorded as to agreement or non-agreement. Labels will be checked for notation of proper preservation. If there is an apparent non-agreement in the document or incorrect preservation noted, the apparent problem will be recorded and the ENTACT QA/QC Officer notified. The samples will then be marked or labeled with laboratory sample numbers. Laboratory project numbers are assigned serially, with each sample numbered as a subset of the project number. Finally, samples will be placed in appropriate storage and/or secure areas.

5.3 FINAL EVIDENCE FILES

The final evidence file will be the central repository for all documents that constitute evidence relevant to sampling and analysis activities as described in this QAPP. ENTACT is the custodian of the evidence file and maintains the contents of the evidence files for the removal action, including all relevant reports,

records, logs, field notes, pictures, and data reviews in a secured, limited access area under the custody of the ENTACT Project Manager.

ENTACT
Section
six

6.0 CALIBRATION PROCEDURES AND FREQUENCY

Procedures described in this section pertain to the calibration, maintenance, and operation of equipment and instrumentation to be used during the implementation of the RA. A variety of instruments, equipment, and sampling tools will be used to collect data and samples to evaluate and monitor Site conditions. Proper calibration, maintenance, and use of instruments and equipment are imperative to ensure the quality of all data collected. A record of calibration and maintenance activities is important to provide legally dependable data.

Instruments and equipment used to gather, generate or measure environmental and physical testing data will be calibrated with sufficient frequency and in such a manner that accuracy and reproducibility are consistent with the manufacturer's specifications.

6.1 FIELD INSTRUMENT CALIBRATION

Field instruments that will be used during this project include XRF equipment. All instruments and equipment purchased or used for the removal action will be inspected to ensure that the item meets and performs to manufacturer's specifications and project specifications. Instruments meeting these requirements will be issued to a field technician trained in operation of the instrument.

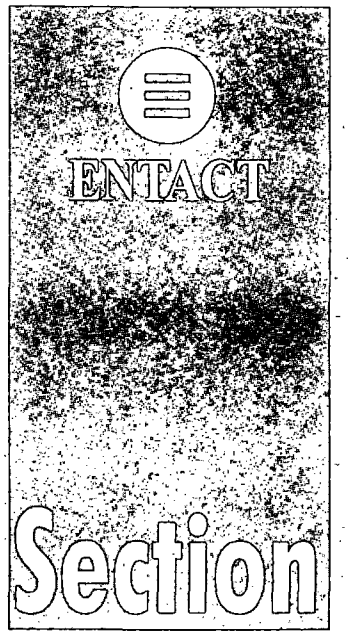
The XRF will be calibrated with the manufacturer's known standards and Site-specific standards. A record of the instrument calibration will be maintained in a bound field notebook and these records will be subject to a QA audit. Information recorded will include the following:

- Date of calibration
- All data pertaining to the calibration procedures
- Initials of analyst performing calibration
- Adjustments made to equipment prior to and following calibration; and
- Record of equipment failure

Any items found to be inoperable will be taken out of use and a note stating the time and date of this action will be made in the calibration logs. The reason for equipment failure and the time and date of its return to service will also be noted in the logbook. Records produced shall be reviewed, maintained, and filed by the field operators. The ENTACT QA Manager will audit these records to verify complete adherence to these procedures.

6.2 LABORATORY INSTRUMENT CALIBRATION

All laboratory instrument calibration procedures can be found in the attached SOPs (Attachment QAPP-C).



Section seven

7.0 ANALYTICAL AND MEASUREMENT PROCEDURES

The laboratory that will be performing sample analysis for this project is:

Severn Trent Laboratories
13715 Rider Trail North
Earth City, Missouri 63045
Phone (314) 298-8566

Complete list of analytical parameters, methods, matrices, holding times and preservation requirements are included in Attachment QAPP-D

7.1 FIELD ANALYTICAL PROCEDURES

Field analytical and test procedures include the following:

Soil
XRF - Total Arsenic, Lead, Zinc, Cadmium

The SOP for this device is located in Attachment QAPP-F.

7.2 LABORATORY ANALYTICAL PROCEDURES

Laboratory analytical test procedures include the following:

Soil:

Total RCRA Metals and Zinc- Method ILMO3.0

Waste Characterization Soils:

TCLP lead - Method 1311/6010

Off-Site Backfill Source:

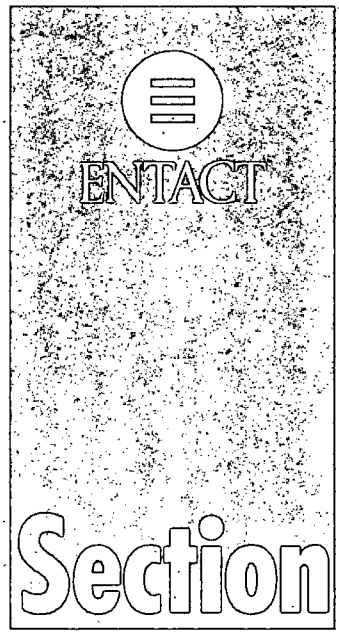
TAL Total Metals – Method 6010/7471
VOCs – Method 8260
SVOCs – Method 8270
Pesticides – Method 8081
PCBs – Method 8082
Extractable Petroleum Hydrocarbon – Method 8015

Air Transport Monitors:

Settleable particulate matter and deposition rate – Method D 1739-98, Standard Test Method for Collection and Measurement of Dust Fall (Settleable Particulate Matter)

7.3 LIST OF TARGET COMPOUNDS AND LABORATORY REPORTING LIMITS

The reporting limits are given in the STL Reference Data Summaries for the analyses required during the RA. The Reference Data Summary Tables can be found in Attachment QAPP-B (B1 through B10)


ENTACT
Section
eight

8.0 QUALITY CONTROL CHECKS

Internal QC procedures are designed to ensure and document the overall quality of data. Two types of QC checks will be employed to evaluate the performance of the laboratory's analytical procedures. The QC checks represent the system checks and controlled samples introduced into the sample analysis stream that are used to validate the data and calculate the accuracy and precision of the chemical analysis program.

Project QC checks are accomplished by submitting controlled samples into the laboratory from the field. Two external types of QC samples will be used: blanks and duplicates. A duplicate sample will be collected for every 10 samples per matrix or one duplicate per day, whichever is greater. Any samples submitted as "blind" samples will be noted in the field logbook and given a sample number that does not indicate to the laboratory that the sample is a QC check.

8.1 FIELD QUALITY CONTROL CHECKS

For field XRF soil analyses, a laboratory sample will be sent to the laboratory for confirmatory total RCRA metals and zinc analysis for ten percent of the field XRF samples. Table QAPP-3 presents the QA criteria for field measurements.

8.2 LABORATORY QUALITY CONTROL CHECKS

Laboratory QC checks are accomplished through the use of system checks and QA/QC samples that are introduced into the same analysis stream. Laboratory system checks and QA/QC samples for inorganics are defined below.

- Calibration Blank - A volume of acidified de-ionized water.
- Continuing Calibration - Analytical standard run every 10 analytical samples or every two hours, whichever is more frequent, to verify the calibration of the analytical system.
- Instrument Calibration - Analysis of analytical standards for a series of different specified concentrations used to define the quantitative response, linearity, and dynamic range of the instrument to target compounds.
- Preparation Blank - An analytical control that contains deionized water and reagents, carried through the entire analytical procedures. An aqueous method blank is treated with the same reagents as a sample with a water matrix; a solid method blank is treated with the same reagents as a soil sample.

Laboratory QA/QC checks will be performed and samples will be analyzed at a frequency established by appropriate SW-846 protocols for inorganic compounds and appropriate SOPs for analytical methods. Attachment QAPP-C defines all the STL Laboratory QC checks criteria for this project. Any QC checks that do not meet acceptance criteria will be handled as discussed in Section 13.0 of the QAPP.

Table QAPP-3
FIELD QC CRITERIA

PARAMETER	METHOD[1] REFERENCE	PRECISION[2]	ACCURACY[2]	COMPLETENESS
SOIL				
Field XRF	Per ENTACT SOP	+ 10%	N/A[3]	90%

NOTES:

1. Methods: E - *Method for Chemical Analysis for Water and Wastes* (U.S. EPA, 1983).
   SW-xxxx – *Methods for the Analysis of Solid Waste (SW-846).*
2. Acceptable accuracy and precision based on the range of measurement. The XRF will be used for field analysis with 10% of the samples submitted to the approved laboratory to verify the accuracy of the XRF results.
3. NA - Not Applicable

ENTACT
Section
nine

9.0 DATA REDUCTION, VALIDATION AND REPORTING

All data collected will be managed, distributed, and preserved to substantiate and document that data are of known quality and are properly maintained. Technical data will be tracked and validated to monitor the performance of the tasks. An outline of the QC data handling process for data collection, transfer, validation, reduction, reporting, and storage for both field and laboratory QC data is described below. The ENTACT QA Manager is responsible for ensuring these tasks are completed.

9.1 DATA REDUCTION

Data quality and utility depends on many factors, including sampling methods, sampling preparation, analytical methods, quality control, and documentation. Once all physical and chemical data are validated and assembled, these data are further evaluated with respect to PARCC parameters. Satisfaction of these criteria will be documented as listed below. Chemical data must meet criteria of (1) quantitative statistical significance, (2) custody and document control, and (3) sample representativeness. Physical data must meet criteria of (1) sampling location, time, and personnel; (2) documentation; and (3) methodologies.

To determine the quantitative statistical significance of chemical data, the following items will be documented as appropriate:

- Laboratory/field instrumentation, including calibration data, standard methods, and references;
- Proper sample bottle preparation;
- Laboratory analysis detection limits;
- Analysis of laboratory (reagent) blanks at a frequency of at least one per 20 samples per matrix;
- Analysis of laboratory spikes at a frequency of at least 1 per 20 samples or one per analytical batch;
- Analysis of field replicates (duplicates or splits) at a frequency of at least 1 per 10 samples for each matrix or one per day, whichever is greater;
- Analysis of laboratory replicates (duplicates or splits) at a frequency of at least 1 per 20 samples; and
- Presentation of tabulated QC data.

To evaluate the custody and document control for samples and results, the following items will be documented:

- Field custody noted in field logbook or COC documentation available;
- Samples transported via courier or hand-delivered to laboratory with COC documentation available;
- Laboratory custody documented by COC documentation from either field personnel or shipper;
- Laboratory custody documented through designated laboratory sample custodian with secured sample storage area;
- Sample designation number(s) traceable through entire laboratory monitoring system;
- Field notebooks and all custody documents stored in secure repository or under the control of a document custodian;
- All forms filled out completely in indelible ink without alterations except as initials;
- Identity of sampler; and
- Date of sample collection, shipping, and laboratory analysis.

To determine sample representativeness the following items must be checked:

- Compatibility between appropriate field and laboratory measurements or suitable explanation of discrepancy;
- Analysis within holding time limits suitable for the preservation and analysis methods used;
- Sample storage within suitable temperature, light, and moisture conditions;
- Proper sample containers used;
- Proper sample collection equipment used and properly decontaminated;
- Proper sample preservation;
- Proper laboratory preparation techniques used;
- An evaluation of factors to determine bias screening; and
- Sample Site selection criteria to provide representativeness.

To evaluate the field physical data that support the analytical data, the following items will be documented:

- Sampling date and time;
- Sampling personnel;
- Sampling location;
- Physical description of sampling location;
- Sample collection technique;
- Field preparation techniques;
- Visual classification of sample using an accepted classification system;
- A thorough description of the methodology used and a rationale for the use of that methodology;
- Complete documentation of record-keeping practices;
- Field notebook and all custody documents stored in a secure repository or under the control of a document custodian; and
- All forms filled out in indelible ink without alterations except as initialed.

9.1.1 Field Data Reduction Procedures

Field data reduction is not anticipated for this project. The data will be generated from direct readout instruments. The data is then downloaded by RS-232 computer port to a database spreadsheet. The field XRF values will be entered into the field logbook so data transcription errors can be discerned easily upon validation. The information will be entered into the field logbook and checked for transcription errors by the sampling team.

9.1.2 Laboratory Data Reduction Procedures

Reduction procedures in the laboratory will be performed by computer database that will provide printouts of raw data and chromatograms. The information will be evaluated by the bench analyst to ensure proper integration and assignment of various sample constituents. Lab records will note all other information not processed by computer such as reagents, sample preparations, etc.

The department supervisor will review the lab notebook and associated computer printouts to ensure all information is accurate and no errors have occurred. Prior to laboratory release of the data, QA/QC will be performed to assess precision and accuracy requirements of the data have been met.

9.2 DATA VALIDATION

Technical data, including field data and results of laboratory sample analyses, will be validated to monitor the performance of the RA. The data collection and quality assurance procedures for validating field and laboratory data are described below.

Field precision is assessed through the collection and measurement of field duplicates at a rate of 1 duplicate per 10 investigative analytical samples.

9.2.1 Procedures Used to Validate Field Data

The ENTACT QA Manager will perform validation of data obtained from field measurements. Such validation will be performed by regularly checking procedures utilized in the field and comparing the data to previous measurements. Data that cannot be validated will also be documented.

Field data requiring validation includes the raw data and supportive documentation generated from field investigations and will include, but is not limited to, the following:

- Field notebooks;
- Field investigation daily reports;
- Field instrument readings and calibration data sheet;
- Field log borings;
- Sample labels;
- COC forms;
- Sample tracking records;
- Surveying information; and
- Maps.

Field measurements that could affect the quality of the data (such as soil water content) will also be validated. Validation of all field data will be performed in terms of meeting DQOs by checking the procedures utilized in the field and comparing the data to previous measurements. The following areas will be addressed during validation:

- Sampling methodology;
- Sample holding times and preservation;
- Field instrument selection and use;
- Field instrument calibration and standardization;
- Field instrument preventative and remedial maintenance;
- Field deviations; and
- Units of measure and reference points from which field data will be measured.

Additional specific evaluations of data critical to the integrity of the decision making process for this task will be performed on 10 percent of the data and will include:

- COC integrity check;
- Review of the appropriateness of field methodologies;
- Transcription, calculation, completeness, and accuracy check of field data; and
- Analysis of field notes to determine presence of bias.

If substantial errors are detected which impact data quality, the scope of the validation will be increased to determine the extent of the problems.

9.2.2 Procedures Used to Validate Lab Data

Under the direction of the Laboratory QA Manager, lab data will be reviewed to ensure that results for samples meet all method-specified criteria. The requirements to be checked in validation are:

- Sample Holding Times
- Calibration
- Blanks
- Matrix Spike/Matrix Spike Duplicate
- Field Duplicate
- Target Compound Identification
- Spectral Interference Check Sample Analysis
- Compound Quantitation and Reported Detection Limits
- System Performance
- Overall Assessment of Data
- Interference Check Sample Analysis
- Laboratory Control Sample Analysis

One equipment blank will be prepared and documented for every 10 investigative samples to assess the accuracy of sampling techniques. One matrix spike and matrix spike duplicate will be analyzed for every 20 investigative samples.

The laboratory QA Manager will be responsible for assessing data quality and advising appropriate laboratory section supervisors of any data that are "unacceptable" or have notations that would caution the data user to possible unreliability. Data reduction, validation, and reporting by the laboratory will be conducted as follows:

- Raw data produced by the analyst will be turned over to the respective supervisor.
- The supervisor will review the data for attainment of QC criteria as outlined in method protocols and established EPA methods.
- Upon completion of analytical testing, the laboratory Project Manager conducts a final review.
- Upon acceptance of the data by the laboratory Project Manager, a computerized report will be generated and sent to the ENTACT QA Manager.
- The ENTACT QA Manager will complete a thorough audit of all reports.

The ENTACT QA Manager will conduct an evaluation of data reduction and reporting by the laboratory. These evaluations will consider the finished data sheets, calculation sheets, document control forms, blank data, duplicate data, and recovery data for matrix and surrogate spikes. The material will be checked for legibility, completeness, and the presence of necessary dates, initials, and signatures. The results of these checks will be assessed and reported, noting any discrepancies and their effect upon acceptability of the data. In addition, the QA Manager will check for data consistency by assessing comparability of duplicate analyses, comparability to previous criteria, transmittal errors, and anomalously high or low parameter values. The results of these checks will be reported in writing.

The following is a description of the validation steps that will be used by the ENTACT QA Manager to validate the laboratory data. These validation results will be summarized in the Final Report on Removal Action. The validation steps are as follows:

- Compile a list of all samples;
- Compile a list of all QC samples;
- Review laboratory analytical procedures and instrument performance criteria.

Specific evaluations critical to the integrity of the data include:

- Review of COC documents for completeness and correctness;
- Transcription, calculation, completeness, and accuracy check; and
- Review of laboratory analytical procedures, appropriateness, and instrument performance criteria.

In addition, data validation will be performed on 10 % of the laboratory confirmation soil sample data, as consistent with approved EPA protocol at previous Superfund projects conducted by ENTACT in Illinois. If significant errors that affect data quality are detected, the percentage of raw data validated will be increased to assess the magnitude of the problem.

A data summary will be prepared and will include:

- Results;
- Sample media identification;
- Sample location and description;
- Appropriate concentration units;
- Appropriate significant figures;
- Data qualifiers; and
- Definitions.

The laboratory data summary will be reviewed for potential data quality problems, including:

- Unexpected results;
- Common laboratory contaminants;
- Samples in which dilution was necessary; and
- Time and date of sample collection.

A sample data summary will be prepared to assess precision, accuracy, and completeness of the analytical

data. Laboratory records and data package requirements will be checked to assess completeness of the data package. The validation effort will be done by personnel qualified and experienced in the field of laboratory data validation.

Despite all efforts to achieve the objectives of the project, the potential for error exists in laboratory chemical analyses and in the data reporting process. Every reasonable effort will be made to compare and double-check data reported from the laboratory with data entered into the data base management system.

9.3 DATA REPORTING

Data generated during the removal activities will be appropriately identified, validated, and summarized in monthly progress reports, and included in the final report. The ENTACT QA Manager will develop a data storage and information system to facilitate and manipulate data for tracking, data calculations, and transfer of data to various forms and reports and transmittal of data into a data storage system. Data packages from the laboratory will be in the form of a Level 3 QC package excluding a sample traffic report and electronic deliverables.

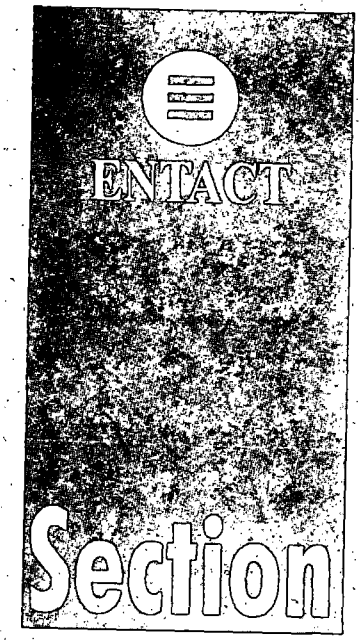
After data validation and reduction, the ENTACT QA/QC Officer will summarize the data obtained and include the information in the field activity report submitted to the Project Manager for review. The ENTACT Project Manager will then prepare monthly reports and the final report for submission to the EPA OSC. The appropriate documents will be prepared and distributed to the appropriate parties to summarize both the field activities performed and the results obtained. The field reports will include presentation of results, summaries of field data from field measurements, and field location of sampling points. All other information will be bound in the appendices. The laboratory reports will include at a minimum the following components:

- Date of issuance;
- Any deviations from the intended analytical strategy;
- Laboratory batch number;
- Number of samples and respective matrices;
- Project name and number;
- Condition of samples;
- Discussion of holding times;
- Discussion of technical problems or observations;
- Discussion of quality control checks which failed;
- Sample description information;
- Analytical tests assigned;
- Analytical results;
- Quality control reports;
- Description of analytical methodology;
- Description of QC methodology; and
- Signature of Laboratory Operations Manager.

Both the field and laboratory reports will contain the following:

- Any changes in the QA Project Plan;

- Significant QA problems, recommended solutions, and results of corrective actions;
- Discussions of whether the QA objectives were met, and the resulting impact on decision making; and
- Limitations on the use of the measurement data.


ENTACT
Section
ten

10.0 PERFORMANCE AND SYSTEMS AUDITS

Two types of audit procedures will be used to assess and document performance and project staff: system audits and performance audits. These audits are performed at frequent intervals under the direction of the ENTACT QA Manager to evaluate quantitatively the accuracy of the total measurement system. These audits form the basis for corrective action requirements and provide a permanent record of the conformance of measurement systems to QA requirements.

System audits consist of quantitative evaluation of field and laboratory quality control measurement systems to determine if they are being used appropriately. These audits may be carried out before all systems are operational, during the program, or after the completion of the program. These audits involve a comparison of the activities presented in the QA plan with those actually scheduled or performed.

Performance audits are a quantitative evaluation of the measurement systems of the program. They require testing of the measurement systems with samples of known composition or behavior to evaluate precision and accuracy after systems are operational and generating data. Analytical laboratories designated to perform analytical services during the RA at the Site may be audited prior to sample analysis.

10.1 INTERNAL AUDITS

A systems audit will be performed on laboratory, office, and field operations prior to or shortly after systems are operational. The system audit protocols are summarized as follows:

Laboratory Operations: Laboratory QA Manager
- Parameter and/or laboratory notebooks;
- Instrument/equipment logbook;
- Sample log-in, routing, and labeling for analysis;
- Updating of QC criteria for spike recoveries; and
- QA Manager will monitor analyses to assure complete adherence to approved analytical methods.

Field Operations: ENTACT QA Officer
- Field notebooks, procedures, field logs, etc.
- Site safety;
- Sampling methods; and
- Sample labeling, packing, storage, shipping, and COC procedures.

Office Operations: ENTACT Field Project Manager
- Project team members are informed of the team organization and in particular the quality control procedures for their work assignment; and
- QC officers assigned to the project are available and informed of the QC they are responsible for, and the schedule for QC review.

After systems are operational and generating data, a performance audit will be conducted at least once during the laboratory, office, and field work to determine the accuracy of the total measurement systems or component parts thereof. The performance audit protocol is summarized as follows:

Laboratory Operations: Laboratory QA Manager
- Sample log-in, routing, and labeling for analysis;
- Analyses to assure complete adherence to approved test methods; and
- Other QC procedures outlined herein.

Field Operations: ENTACT QA Officer
- Field notebooks, procedures, field logs, etc.
- Site safety;
- Sampling methods; and
- Sample labeling, packing, storage, shipping, and COC procedures.

Office Operations: ENTACT Field Project Manager
- Specified QC reviews of the work are being performed;
- The individuals performing the QC reviews are qualified and as assigned; and
- Final reports and deliverables have received the appropriate QC review.

The auditor will maintain a record of the evaluation by writing field notes. Following the audit, the preliminary results will be reviewed with the person in charge of the operations audited. Subsequent to the audit, the auditor will develop an audit report that summarizes the areas requiring corrective measures. This report will be submitted to the ENTACT Project Manager.

10.2 EXTERNAL AUDITS

In addition to these internal field and laboratory audits, the EPA Region 5 QA reviewer may conduct external field and laboratory audits. The EPA Project Coordinator may also perform external field and laboratory audits. The external field audits may be conducted any time during the field operations and may or may not be announced. An external audit may be performed at least once prior to the initiation of the sampling and analysis activities. These audits may or may not be announced. The external lab audit will include (but not be limited to) review of laboratory procedures, laboratory on-Site audits, and/or submission of performance verification samples to the laboratory for analysis.

ENTACT
Section
eleven

11.0 PREVENTATIVE MAINTENANCE

To minimize the occurrence of instrument failure and other system malfunction, a preventative maintenance program for field and laboratory instruments will be implemented. Equipment, instruments, tools, gauges, and other items requiring preventative maintenance will be serviced in accordance with the manufacturer's specified recommendations and written procedures developed by the operators. A person certified to repair the instrument would perform maintenance items that cannot be performed by the laboratory technician. The laboratory will be responsible for performing routine maintenance and will have available tools and spare parts to conduct routine maintenance. Two backup XRF units will be available for use in the case of a malfunction to avoid downtime.

Manufacturer's procedures identify the schedule for servicing critical items in order to minimize the downtime for the measurement system. It will be the responsibility of the field instrument operator and the laboratory to adhere to this maintenance schedule and arrange any necessary and prompt service. In addition to any manufacturer recommended maintenance criteria, a maintenance procedure will be developed by the operator based upon experience and previous use of the equipment. Service to the equipment, instruments, tools, gauges, etc., shall be performed by qualified personnel. Periodic maintenance is shown on Table QAPP-4.

Logs are used to record maintenance and service procedures and schedules. All maintenance records will be documented and traceable to the specific equipment, instruments, tools, and gauges. Any items found to be inoperable will be taken out of use and a note stating the time and date of this action will be made in the calibration sheets and logs. The reason for equipment failure and the time and date of its return to service will also be noted in the logbook. Records produced shall be reviewed, maintained, and filed by the operators at the laboratories and by the data and sample control personnel when and if equipment, instruments, tools, and gauges are used at the Site. The ENTACT Project Manager will audit these procedures.

Table QAPP-4
Maintenance Procedures for Field and Laboratory Equipment

Instrumentation	Maintenance Procedure	Spare Parts
Field XRF	1. Leak testing every six months 2. Shutter check every six months 3. Annual manufacturer servicing	Battery packs XRF Cables
Gas Chromatograph/Mass Spectrometer	1. Change septa as needed 2. Change syringes on autosamplers as needed 3. Leak check when installing columns 4. Injection port cleaning as needed 5. Check inlet system for residue buildup periodically 6. Clean gas line dryers as needed 7. Replace pump oil as needed 8. Replace electron multiplier as needed	Syringe Septa Various electronic components Plumbing supplies Injection port liners
ICP Spectrometer	1. Change sample rinse lines 2. Clean nebulizer components and torch assembly 3. Clean filters 4. Clean mirrors	Nebulizer components Torch assembly Pump tubing Sample probe

ENTACT
Section
twelve

12.0 SPECIFIC ROUTINE PROCEDURES USED TO ASSESS DATA PRECISION, ACCURACY, AND COMPLETENESS

This section summarizes the QA/QC procedures used in assessing the quality of the chemical data and the format for presenting the results of the QA/QC evaluations. The data evaluation procedures will be used by the QA Manager for assessing duplicate and spike samples and checking blank samples that are submitted blind to the analytical laboratories from the field or generated internally by the laboratory, in accordance with this QAPP. The purpose of implementing these procedures is to assess the chemical data generated for accuracy, precision, representativeness, and completeness for both the laboratory analytical program and field sample collection activities.

The primary goal of the program is to ensure that the data generated are representative of environmental conditions at the Site. Accuracy, precision, representativeness, and completeness will be computed in the manner described in the following paragraphs. A qualitative assessment of accuracy, precision, representativeness, and completeness will be made and documented. The goal of the assessment will be to (1) establish Site specific PARCC parameters; (2) use the parameters to develop a database with known limitations of data usability; and (3) evaluate these limitations in achieving the project DQOs. Complex statistical data verification and a significance evaluation will not be performed. If a problem arises and the data are found to deviate from previous analyses or surrounding conditions, the data will be annotated. Sample recollection and analysis will be used only in extreme cases of QC problems.

Chemical data will be evaluated according to accuracy, precision, representativeness, and completeness criteria for both the field sample collection activities and laboratory analytical programs. The QA/QC program will evaluate data based on three types of quality control samples (matrix spikes, blanks, and duplicates).

The completeness of the data represents the amount of valid data obtained from the field programs versus the amount of data expected under normal conditions. Completeness will be assessed prior to preparation of the final report. These procedures for evaluating the field and laboratory QA/QC data are the same and are presented below for QA/QC matrix spike, blank, and duplicate samples.

12.1 ACCURACY ASSESSMENT

In order to assure the accuracy of the analytical procedures, an environmental sample is randomly selected from each sample shipment received at the laboratory, and spiked with a known amount of the analyte to be evaluated. In general, a sample spike should be included in every set of 20 samples tested on each instrument. The spike sample is then analyzed. The increase in concentration of the analyte observed in the spiked sample, due to the addition of a known quantity of the analyte, compared to the reported value of the same analyte in the unspiked sample determines the percent recovery. The percent recovery for a spiked sample is calculated according to the following formula:

$$\% \text{ Recovery} = \frac{\text{Amount in spiked sample - Amount in sample}}{\text{Known amount added}} \times 100$$

12.2 PRECISION ASSESSMENT

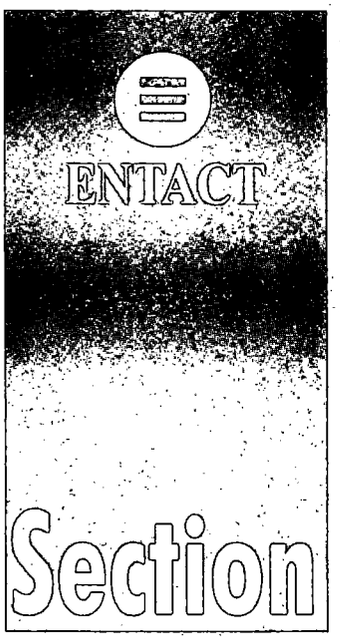
Spiked samples are prepared by choosing a sample at random from each sample shipment received at the laboratory, dividing the sample into equal aliquots, and then spiking each of the aliquots with a known amount of analyte. The duplicate samples are then included in the analytical sample set. The splitting of the sample allows the analyst to determine the precision of the preparation and analytical techniques associated with the duplicate sample. The relative percent difference (RPD) between the spike and duplicate spike are calculated and plotted. The RPD is calculated according to the following formula:

$$RPD = \frac{\text{Amount in Spike 1 - Amount in Spike 2}}{0.5\ (\text{Amount in Spike 1} + \text{Amount in Spike 2})} \times 100$$

12.3 COMPLETENESS ASSESSMENT

Completeness is the ratio of the number of valid sample results to the total number of samples analyzed with a specific matrix and/or analysis. Following completion of the analytical testing, the percent completeness will be calculated by the following equation:

$$\text{Completeness} = \frac{\text{(Number of valid measurements)}}{\text{(Number of measurements planned)}} \times 100$$



Section thirteen

13.0 CORRECTIVE ACTION

The following procedures have been established to assure that conditions adverse to quality, such as malfunctions, deficiencies, deviations, and errors, are promptly investigated, documented, evaluated, and corrected. When a significant condition adverse to quality is noted at the Site or laboratory, the cause of the condition will be determined and corrective action taken immediately. All project personnel have the responsibility to promptly identify, solicit approved correction, and report conditions adverse to quality. Conditions, which warrant corrective action, include:

- Predetermined acceptance standards are not attained;
- Procedures or data compiled are determined to be faulty;
- Equipment or instrumentation is found to be faulty;
- Samples and test results are questionably traceable;
- Quality assurance requirements have been violated; and
- System and performance audits indicate problems.

13.1 FIELD CORRECTIVE ACTION

The need for corrective action will be identified as a result of the field audits previously described. If problems become apparent that are identified as a problem originating in the field, immediate corrective action will take place. If immediate corrective action does not resolve the problem, appropriate personnel will be assigned to investigate and evaluate the cause of the problem. When a corrective action is implemented, the effectiveness of the action will be verified such that the end result is elimination of the problem.

Corrective action in the field may be needed when the sampling network is changed, sampling procedures are changed, or field analytical procedures require modification due to unexpected conditions. In general, the Project Coordinator, QA/QC Officer, QA Manager, and Field Project Manager may identify the need for corrective action. The ENTACT field staff in consultation with the ENTACT Project Manager will recommend the corrective action. The ENTACT Project Manager will approve the corrective measure, which will be implemented by the ENTACT Field Team. It will be the responsibility of the ENTACT Project Manager to ensure that corrective action has been implemented.

If the corrective action will supplement the existing sampling plan using existing and approved procedures in the QAPP, corrective action approved by the ENTACT Project Manager will be documented. If corrective actions result in fewer samples, alternate locations, etc. which may cause project QA objectives not to be achieved, it will be necessary for all levels of project management, including EPA to concur with proposed changes.

Corrective action resulting from internal field audits will be implemented immediately if data may be adversely affected due to unapproved or improper use of approved methods. The ENTACT QA Manager will identify deficiencies and recommended corrective action to the ENTACT Project Manager. Implementation of corrective actions will be performed by the ENTACT Field Team. Corrective action will be documented in QA reports to the entire project management. The EPA will be notified immediately if any problems affecting data quality occur.

Corrective actions will be implemented and documented in the field record book. No staff member will initiate corrective action without prior communication of findings through the proper channels. If corrective actions are insufficient, the EPA OSC may suspend work.

13.2 LABORATORY CORRECTIVE ACTION

The laboratory QA/QC Manager in consultation with the Laboratory Operations Manager will initiate the need for corrective action resulting from QA audits. The corrective action will be performed prior to the release of data from the laboratory. The corrective action will be documented in the logbook and submitted to the data validator. If the corrective action does not rectify the situation, the laboratory will contact the ENTACT Project Manager. If the nonconformance causes project objectives not to be achieved, it will be necessary to inform all levels of ENTACT management and the EPA. Corrective action may include, but is not limited to:

- Reanalyzing the samples, if holding time criteria permit;
- Evaluating and amending sampling and analytical procedures;
- Accepting data with an acknowledged level of uncertainty; and
- Resampling and analysis, if the completeness of the data set or intended use of the data is recognized during a preliminary review to be insufficient to meet program DQOs.

If the above corrective actions are deemed unacceptable, an alternate laboratory will be selected to perform necessary analyses.

13.3 CORRECTIVE ACTION DURING DATA VALIDATION AND DATA ASSESSMENT

The facility may identify the need for corrective action during either the data validation or data assessment. Potential types of corrective action may include resampling by the field team or reinjection/reanalysis of samples by the laboratory. These actions are dependent upon the ability to mobilize the field team, and whether the data to be collected is necessary to meet the required QA objectives (e.g. the holding time has not been exceeded, etc.). The ENTACT QA Manager is responsible for identifying a corrective action situation, documenting the incident, determining the course of action, and implementing the corrective action.

13.4 IMMEDIATE CORRECTIVE ACTION

Any equipment and instrument malfunctions will require immediate corrective actions. The laboratory QC charts are working tools that identify appropriate immediate corrective actions to be taken when a control limit has been exceeded. They provide the framework for uniform actions as part of normal operating procedures. The actions taken should be noted in field or laboratory logbooks. A detailed description of method-specific corrective action limits is provided in the appropriate method. The ENTACT QA Manager must approve any deviation from the prescribed control limits in writing.

13.5 LONG-TERM CORRECTIVE ACTION

The need for long-term corrective action may be identified by standard QC procedures, control charts, and system audit. Any procedural or data quality problem that cannot be solved by immediate corrective

action becomes a long-term corrective action. The essential steps in a corrective action system are as follows:

- Identification and definition of the problem;
- Investigation and determination of the cause of the problem;
- Determination and implementation of a corrective action to eliminate the problem; and
- Verification that the corrective action has eliminated the problem.

Documentation of the problem is important in corrective action. The responsible person may be an analyst, ENTACT QA Manager, laboratory QA Manager, sampler, or the ENTACT Project Manager. In general, the designated QA Manager will investigate the situation and determine who will be responsible for implementing the corrective action. The QA Manager will verify that the corrective action has been taken, appears effective, and that the problem has been resolved.

The required corrective action will be documented by the ENTACT QA Manager and the ENTACT Project Manager for field activities. The corrective action will be discussed with the ENTACT Project Manager and the EPA OSC prior to implementation if the severity of the problem warrants such discussion.

Any changes proposed for amending sampling and analytical procedures will be approved by the EPA prior to implementation. These changes will be documented in monthly progress reports and addenda to the QAPP.

Project management and staff, including field investigation teams, document and sample control personnel, and laboratory groups, will monitor on-going work performance in the normal course of daily responsibilities. The ENTACT Project Manager will monitor work at the Site.

Following identification of an adverse condition or QA problem, the ENTACT QA Manager will notify the ENTACT Project Manager of the problem.

ENTACT
Section
fourteen

14.0 QUALITY ASSURANCE REPORTS TO MANAGEMENT

14.1 CONTENTS OF A PROJECT QA REPORT

Analytical results of samples analyzed during the RA will be submitted to the Project Manager following a QA/QC review. The results will include a tabulation of the analytical data and an explanation of any field conditions or laboratory QA/QC problems and their effects on data quality. Results of performance audits and system audits will also be included, as appropriate. Proposed corrective action will be recommended in the event that QA problems are identified during review of data quality or results of performance or system audits.

The final report will contain a discussion of QA/QC evaluations summarizing the quality of the data collected and/or used as appropriate to each activity of the project. The objective of the QA/QC summary will be to ensure that the data are representative of Site conditions and sufficient in quality and quantity to support the field activities. The QA/QC summary will include:

- Tabulated results of all field and analytical data;
- A report from the laboratory QA Manager evaluating the validity of the analytical data with respect to accuracy, precision, completeness, and representativeness; and
- A report from the ENTACT Project Manager evaluating the results of field and office audits; if conducted.

A QA report will be prepared by the QA Manager upon receipt of sufficient QA data from the laboratory. The report will be a summary of QA/QC results of the analytical work conducted and will be included as part of the final RA report.

14.2 QA REPORTING AND ROUTING SCHEDULE

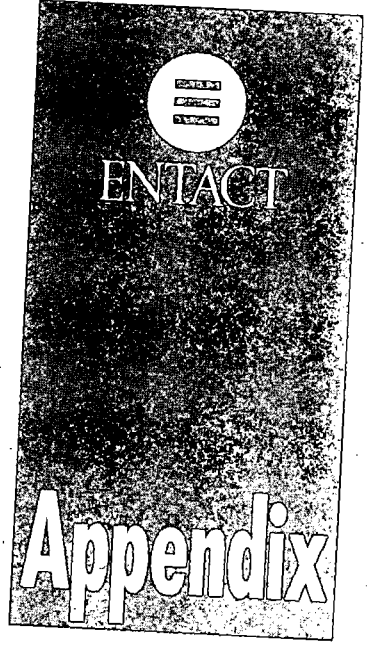
The QA Reports will be prepared on a monthly basis and will be delivered to all recipients by the end of the first full week of the month. The reports will continue without interruption, until the project has been completed. All individuals identified in the Project Organization Chart will receive copies of the monthly QA Report.

QUALITY ASSURANCE PROJECT PLAN
FOR THE
OLD AMERICAN ZINC SITE
Fairmont City, St. Clair County, Illinois
April 2002

Prepared by:

ENTACT & Associates, LLC.
1360 N. Wood Dale Rd.
Wood Dale, Illinois 60191

Rich Wood, ENTACT Project Manager	Date
Caroline Panico, ENTACT Quality Assurance Manager	Date
Gary Uphoff, EMS Project Coordinator	Date
John Powell, Severn Trent Laboratory QA Manager	Date
EPA Superfund QA Reviewer	Date
EPA Remedial Project Manager	Date



Appendix A

ATTACHMENT QAPP-A

ENTACT MANAGEMENT TEAM QUALIFICAITONS

Richard A. Wood

ENTACT

MARKETING/PROJECT COORDINATOR

1994 - Present

Since joining ENTACT in 1994, Mr. Wood has worked on several CERCLA and RCRA sites. His responsibilities have included project management, field QA/QC, air, soil, and water sampling, Health and Safety, project reporting, waste profiling, coordination of transportation and disposal of waste streams, and public relations.

ENTACT EXPERIENCE SUMMARY

Circle Smelter Superfund Site
Beckemeyer, Illinois - CERCLA - Region V – Former Zinc Smelter/Active Residential Area
Coordinated all aspects of project activities at this Superfund Site, including soil and air sampling, excavation, backfill and restoration of over 500 residential lots, public relations with all property owners before, during, and after remedial activities, data management, transportation and on-site disposal of impacted soils and project reporting. Managed remediation of the industrial property including decontamination and demolition of several structures, excavation of 120,000 cubic yards of industrial soils, and construction of a 120,000 square foot on-site repository area to contain residential and industrial impacted soils and debris.

Residential Mercury Cleanup
Chicagoland Area, Illinois – Mercury Impacted Residential Properties
Coordinated all aspects of project activities including the interior sampling, decontamination and clearance sampling of approximately 100 residential properties in the Chicagoland area. Under Mr. Wood's coordination efforts, ENTACT was evaluated as a top three performer out of approximately 16 contractors initially engaged for this Chicago suburban wide emergency cleanup. ENTACT completed the activities for a large Chicagoland utility company.

NL Industries/Taracorp Superfund Site
Granite City, Illinois - CERCLA - Region V - Secondary Lead Smelter/Battery Breaking Facility
Managed all aspects of project activities at this Superfund Site, including soil and air sampling, excavation, backfill and restoration of over 800 residential lots, public relations with all property owners before, during, and after remedial activities, data management, transportation and off-site disposal of lead contaminated soils and project reporting. Managed remediation of the industrial property including excavation of 50,000 cubic yards of industrial soils, backfill of excavated areas, construction of a 60,000 square foot landfill extension, consolidation of excavated materials onto a 90,000 cubic yard slag pile and construction of a 6.5 acre multilayer RCRA cap over the slag and excavated industrial soils.

Avanti Industrial Site, Battery Breaking Facility
Indianapolis, Indiana - CERCLA 106 (a) Unilateral Administrative Order - Region V
Mr. Wood managed all aspects of field quality assurance and quality control during Phase I project activities at this CERCLA 106 Emergency Removal Action, including soil and air sampling, utilization of a field X-ray fluorescence spectrum analyzer to guide excavation lead contaminated soils on 256 residential properties, public relations with all property owners before, during, and after remedial activities, data management and development of the Phase I Final Report.

David Hinton

ENTACT

Field Project Manager

1997 - Present

Mr. Hinton is responsible for the management of field aspects of ENTACT projects. Some obligations include work plan preparation, implementation of field construction activities, heavy equipment operation and coordination, equipment maintenance and field crew leadership. He has been involved in numerous site walks and investigations and has helped develop innovative and cost effective solutions for numerous ENTACT projects.

ENTACT EXPERIENCE SUMMARY

NL Industries/Taracorp Superfund Site, Secondary Lead Smelter/Battery Breaking Facility
Granite City, Illinois - CERCLA - Region V
Responsible for field project management of activities at this NPL site. Scope of work included excavation of lead impacted materials from 720 residential properties, backfill of properties and full restoration. He was also responsible for the consolidation of the lead impacted material for the construction of an on-site cap and containment system.

USS Lead, Battery Breaking Facility and Lead Smelter
East Chicago, Indiana - RCRA 3008 (h) Administrative Order of Consent
Mr. Hinton was responsible for the field implementation of a remedial solution at this former copper refinery and lead smelter. This included extensive building decontamination and demolition, berm construction, lead impacted material excavation (200.000 cubic yards), slag excavation and construction of a 14 acre cap and containment system.

Master Metals, Inc. - Secondary Lead Smelter
Detroit, Michigan - CERCLA 106 Administrative Order
Responsible for the field implementation at this CERCLA 106 Emergency Removal Action. This project included the decontamination and demolition of a secondary lead smelter, on-site treatment of various waste streams, excavation and treatment of on-site soils, asbestos abatement, off-site disposal of PCB's, listed wastes, lab pack, and mercury, waste water treatment and disposal and completion of a non-time critical Engineering Evaluation/Cost Analysis (EE/CA).

Avanti Removal Action, Battery Breaking Facility
Indianapolis, Indiana - CERCLA 106 (a) Unilateral Administrative Order - Region V
Mr. Hinton was responsible for field project management of activities at this CERCLA 106 (a) Administration Order. The scope included excavation of lead impacted materials from 280 residential properties surrounding this former battery breaker and secondary lead smelter. Also, he was responsible for consolidation of lead impacted materials in preparation for an on-site cap and containment system

TRAINING & CERTIFICATIONS

OSHA 29 CFR 1910.120 - 40 Hour Hazardous Materials Training Course
OSHA 29 CFR 1910-120 - Hazardous Material and Emergency Response 8 hour refresher
Confined Space Worker Regulation Title 29CFR 1910.146
X-Ray Fluorescence User Training

Caroline Panico

ENTACT

REGULATORY/TECHNICAL SPECIALIST

1999 - Present

Ms. Panico assists in reviews of regulatory requirements for CERCLA and RCRA sites in preparation for negotiations with state and federal agencies. She assists in the development of remedial strategies that are protective of the environment, meet regulatory requirements, and are cost effective. She also participates in design and product specification review of geosynthetic cell/cover systems.

ENTACT EXPERIENCE SUMMARY

Former Gulf, Mobile and Ohio Rail Yard Site
Murphysboro, Illinois
Provided Administrative Project Management for remedial activities conducted under an Administrative Order by Consent from Region V, USEPA. Remedial activities included excavation, treatment, off site disposal of lead impacted soil and site restoration. Performed project financial control and coordinated business activities with vendors and client. Prepared and submitted reports and documentation required by the USEPA to ensure the performance standards were met throughout the implementation of the remedial activities.

Ramona Park Landfill Battery Casing Area
Utica, Michigan
Provided Administrative Project Management for remedial activities conducted under an Administrative Order by Consent from Region V, USEPA. Remedial activities included excavation, treatment, off site disposal of impacted soil and site restoration. Responsibilities included performing daily, weekly and monthly reporting and communication requirements to project participants. Performed project financial control and coordinated business activities with vendors and client. Prepared and submitted reports and documentation required by the USEPA to ensure the performance standards were met throughout the implementation of the remedial activities.

The Manitowoc Company
Manitowoc, Wisconsin
Provided regulatory assistance in obtaining a Voluntary Party Liability Exemption (VPLE) from the Wisconsin Department of Natural Resources (WDNR) for a project enrolled in the state voluntary cleanup program. Remedial activities included excavation, treatment and off site disposal of impacted soil; site restoration; and groundwater monitoring to evaluate natural attenuation. Interfaced with the client as well as WDNR representatives to guide the project through the state program and authored plans and reports required for submittal purposes.

Tonolli Superfund Site
Nesquehoning, Pennsylvania
Managed the Quality Assurance/Quality Control elements of cleanup activities at this former secondary lead smelter. QA/QC activities included verification of excavation and verification of stabilization of lead impacted material. Also managed QA/QC requirements associated with the construction of a seven-acre RCRA Subtitle C multi-layer cap and leachate collection system.

Jonathan E. Patlak, CHMM

ENTACT

HEALTH AND SAFETY DIRECTOR

2001 - Present

Since joining ENTACT, Mr. Patlak has managed the Health and Safety (H & S) Program for the Chicago office. Corporate responsibilities include: establishing general H & S policies and standards, preparing and amending site specific health and safety plans, approving and managing occupational health care providers and industrial hygiene equipment suppliers, creating and coordinating vehicle fleet safety, conducting intensive H & S training sessions, maintaining associate medical surveillance and training records, review project statistics, recognizing and rewarding associates involved in achieving safety goals, and publishing an in-house quarterly newsletter. Field responsibilities include overseeing site activities for safety compliance, providing regulatory reference support for safety issue resolution, performing safety training and site orientation of field personnel, and conducting site safety audits of project worksites.

ENTACT EXPERIENCE SUMMARY

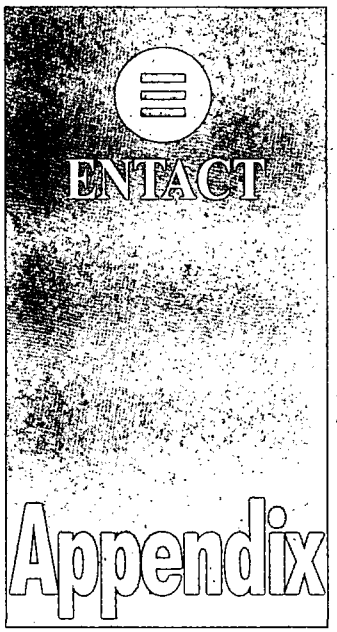
Former Manufactured Gas Plant Site
Oak Park, Illinois
Health and Safety Director for the environmental remediation and restoration of this former Manufactured Gas Plant regulated under the IEPA's Site Remediation Program. Scope of Work includes the excavation and rail transport of approximately 200,000 tons of impacted soils and underground structures, excavation of streets and utility improvements (sewer, gas and water) and restoration of the site. Duties include conducting daily "tailgate" safety meeting, directing extensive interaction with project stakeholders.

Former Stanley Tools Facility
Fowlerville, Michigan
Health and Safety Director for environmental remediation and restoration activities under an Interim Stabilization Measures Action. Project scope includes the excavation of underground piping along the Red Cedar River, sampling, characterization and disposal impacted soils, and restoration of affected areas. Environmental contaminants of concern include TPH and PCBs.

ASARCO Circle Smelting Corporation Site / Beckemeyer Superfund Site
Beckemeyer, Illinois – {CERCLA 106 (a) Unilateral Administrative Order - Region V}
Health and Safety Director for this CERCLA (SACM) Emergency Removal Action, including sampling activities and disposal of 12,000 cubic yards of lead contaminated soil. Removal activities were conducted throughout a village encompassing over 200 residential lots. Over 400 residential properties are currently being remediated with the 75,000 cubic yards of impacted material and an additional 50,000 cubic yards of impacted material being consolidated into a four acre cap and containment system.

Exide Technologies – Former Bowers Battery Site
New Philadelphia, Ohio
Health and Safety Director for this CERCLA site under USEPA Administrative Order by Consent. Scope of Work included the excavation and management of over 6,000 tons lead impacted soils with over 5,000 tons treated on-site prior to off-site disposal.

Union Pacific Rail Road - New Omaha Convention Center/Arena Site
Omaha, Nebraska
Health and Safety Director for the Omaha Shops Corrective Measures Implementation and Interim Action Removals. Scope of Work included the excavation and management of over 100,000 tons of VOC, Lead and Asbestos impacted soils.



Appendix B

ATTACHMENT QAPP-B

SEVERN TRENT LABORATORY REFERENCE DATA SUMMARY

Attachment QAPP-B1

Inductively Coupled Plasma (6010B Trace)

STL Reference Data Summary

Target Analyte List: STL: ICAP TRACE METALS (27)

Matrix: SOLID
Extraction: METALS, TOTAL - Soils
Method: Inductively Coupled Plasma (6010B Trace)
QC Program: STANDARD TEST SET
Location: STL St. Louis

Target List 6043		Detection Limits				Check List 6018					Spike List 6008				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
Aluminum	20	mg/kg	0.85	mg/kg	20011030	9200	mg/kg	58	142	20	200	mg/kg	75	125	20
Antimony	1	mg/kg	0.22	mg/kg	20011030	62.7	mg/kg	27	225	20	50	mg/kg	75	125	20
Arsenic	1	mg/kg	0.096	mg/kg	20011030	47.5	mg/kg	72	126	20	200	mg/kg	75	125	20
Barium	20	mg/kg	0.36	mg/kg	20011030	509	mg/kg	77	123	20	200	mg/kg	75	125	20
Beryllium	0.5	mg/kg	0.018	mg/kg	20011030	55.9	mg/kg	78	122	20	5	mg/kg	75	125	20
Cadmium	0.5	mg/kg	0.022	mg/kg	20011030	157	mg/kg	75	125	20	5	mg/kg	75	125	20
Calcium	500	mg/kg	21.6	mg/kg	20011030	11700	mg/kg	75	125	20	5000	mg/kg	75	125	20
Chromium	1	mg/kg	0.11	mg/kg	20011030	51.4	mg/kg	76	124	20	20	mg/kg	75	125	20
Cobalt	5	mg/kg	0.089	mg/kg	20011030	88.4	mg/kg	78	122	20	50	mg/kg	75	125	20
Copper	2.5	mg/kg	0.26	mg/kg	20011030	69.5	mg/kg	82	118	20	25	mg/kg	75	125	20
Iron	10	mg/kg	3.13	mg/kg	20011030	13700	mg/kg	61	139	20	100	mg/kg	75	125	20
Lead	0.3	mg/kg	0.067	mg/kg	20011030	186	mg/kg	75	125	20	50	mg/kg	75	125	20
Magnesium	500	mg/kg	9.61	mg/kg	20011030	3070	mg/kg	74	126	20	5000	mg/kg	75	125	20
Manganese	1.5	mg/kg	0.036	mg/kg	20011030	674	mg/kg	76	124	20	50	mg/kg	75	125	20
Molybdenum	4	mg/kg	0.11	mg/kg	20011030	62.7	mg/kg	76	123	20	100	mg/kg	75	125	20
Nickel	4	mg/kg	0.13	mg/kg	20011030	112	mg/kg	78	122	20	50	mg/kg	75	125	20
Selenium	0.5	mg/kg	0.20	mg/kg	20011030	109	mg/kg	74	127	20	200	mg/kg	75	125	20
Silver	1	mg/kg	0.068	mg/kg	20011030	84.3	mg/kg	64	135	20	5	mg/kg	75	125	20
Strontium	5	mg/kg	0.12	mg/kg	20011030	89.7	mg/kg	72	127	20	100	mg/kg	75	125	20
Thallium	1	mg/kg	0.19	mg/kg	20011030	66.2	ug/kg	57	143	20	200	mg/kg	75	125	20
Thorium	50	mg/kg	0.80	mg/kg	20011030	100	mg/kg	80	120	20	20	mg/kg	75	125	20
Tin	10	mg/kg	0.64	mg/kg	20011030	119	mg/kg	66	134	20	100	mg/kg	75	125	20
Titanium	5	mg/kg	0.091	mg/kg	20011030	246	mg/kg	36	164	20	100	mg/kg	75	125	20
Uranium	50	mg/kg	1.49	mg/kg	20011030	200	mg/kg	80	120	20	100	mg/kg	75	125	20
Vanadium	5	mg/kg	0.071	mg/kg	20011030	136	mg/kg	68	132	20	50	mg/kg	75	125	20
Zinc	2	mg/kg	0.43	mg/kg	20011030	289	mg/kg	78	123	20	50	mg/kg	75	125	20
Zirconium	10	mg/kg	0.22	mg/kg	20011030						100	mg/kg	75	125	20

Attachment QAPP-B2

Mercury (7471A, Cold Vapor)

STL Reference Data Summary

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: METALS, TOTAL (Method Exclusive) - Solids
Method: Mercury (7471A, Cold Vapor) - Solids
QC Program: STANDARD TEST SET
Location: STL St. Louis

Analyte List	Detection Limits					Check List 6111					Spike List 6112				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
Mercury	0.0333	mg/kg	0.0078	mg/kg	20020209	6.21	mg/kg	68	133	20	0.1667	mg/kg	75	125	20

Attachment QAPP-B3

Base/Neutrals and Acids (8270C)

STL Reference Data Summary

Target Analyte List: STL: SW-846 8270C

Matrix: SOLID
Extraction: SONICATION - Low Level
Method: Base/Neutrals and Acids (8270C)
QC Program: STANDARD TEST SET
Location: STL St. Louis

Target List 6010		Detection Limits				Check List 6151					Spike List 6152				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
Acenaphthene	330	ug/kg	90.57	ug/kg	20020206	1667	ug/kg	14	105	75	1667	ug/kg	10	122	75
Acenaphthylene	330	ug/kg	84.87	ug/kg	20020206	1667	ug/kg	50	150	15	1667	ug/kg	50	150	20
Anthracene	330	ug/kg	86.00	ug/kg	20020206	1667	ug/kg	50	150	15	1667	ug/kg	50	150	20
Benzo(a)anthracene	330	ug/kg	88.85	ug/kg	20020206	1667	ug/kg	50	150	15	1667	ug/kg	50	150	20
Benzo(b)fluoranthene	330	ug/kg	93.52	ug/kg	20020206	1667	ug/kg	50	150	15	1667	ug/kg	50	150	20
Benzo(k)fluoranthene	330	ug/kg	118.20	ug/kg	20020206	1667	ug/kg	50	150	15	1667	ug/kg	50	150	20
Benzo(ghi)perylene	330	ug/kg	132.45	ug/kg	20020206	1667	ug/kg	50	150	15	1667	ug/kg	50	150	20
Benzo(a)pyrene	330	ug/kg	92.18	ug/kg	20020206	1667	ug/kg	50	150	15	1667	ug/kg	50	150	20
bis(2-Chloroethoxy)methane	330	ug/kg	71.61	ug/kg	20020206	1667	ug/kg	50	150	15	1667	ug/kg	50	150	20
bis(2-Chloroethyl) ether	330	ug/kg	50.91	ug/kg	20020206	1667	ug/kg	50	150	15	1667	ug/kg	50	150	20
bis(2-Ethylhexyl) phthalate	330	ug/kg	70.92	ug/kg	20020206	1667	ug/kg	50	150	15	1667	ug/kg	50	150	20
4-Bromophenyl phenyl ether	330	ug/kg	96.85	ug/kg	20020206	1667	ug/kg	50	150	15	1667	ug/kg	50	150	20
Butyl benzyl phthalate	330	ug/kg	79.98	ug/kg	20020206	1667	ug/kg	50	150	15	1667	ug/kg	50	150	20
Carbazole	330	ug/kg	93.12	ug/kg	20020206	1667	ug/kg	50	150	15	1667	ug/kg	50	150	20
4-Chloroaniline	330	ug/kg	64.39	ug/kg	20020206	1667	ug/kg	50	150	15	1667	ug/kg	50	150	20
4-Chloro-3-methylphenol	330	ug/kg	73.68	ug/kg	20020206	1667	ug/kg	14	106	75	1667	ug/kg	11	108	75
2-Chloronaphthalene	330	ug/kg	85.90	ug/kg	20020206										
2-Chlorophenol	330	ug/kg	68.32	ug/kg	20020206	1667	ug/kg	30	118	75	1667	ug/kg	10	107	75
4-Chlorophenyl phenyl ether	330	ug/kg	101.27	ug/kg	20020206	1667	ug/kg	50	150	15	1667	ug/kg	50	150	20
Chrysene	330	ug/kg	106.01	ug/kg	20020206	1667	ug/kg	50	150	15	1667	ug/kg	50	150	20
Dibenzo(a,h)anthracene	330	ug/kg	101.25	ug/kg	20020206										
Dibenzofuran	330	ug/kg	102.10	ug/kg	20020206	1667	ug/kg	50	150	15	1667	ug/kg	50	150	20
Di-n-butyl phthalate	330	ug/kg	88.85	ug/kg	20020206	1667	ug/kg	50	150	15	1667	ug/kg	50	150	20
1,2-Dichlorobenzene	330	ug/kg	34.932	ug/kg	19981104										
1,3-Dichlorobenzene	330	ug/kg	32.79	ug/kg	19981104										
1,4-Dichlorobenzene	330	ug/kg	29.633	ug/kg	19981104										
3,3'-Dichlorobenzidine	1600	ug/kg	80.43	ug/kg	20020206	1667	ug/kg	50	150	15	1667	ug/kg	50	150	20
2,4-Dichlorophenol	330	ug/kg	71.30	ug/kg	20020206	1667	ug/kg	50	150	15	1667	ug/kg	50	150	20
Diethyl phthalate	330	ug/kg	96.60	ug/kg	20020206	1667	ug/kg	50	150	15	1667	ug/kg	50	150	20
2,4-Dimethylphenol	330	ug/kg	66.32	ug/kg	20020206	1667	ug/kg	50	150	15	1667	ug/kg	50	150	20
Dimethyl phthalate	330	ug/kg	93.90	ug/kg	20020206	1667	ug/kg	50	150	15	1667	ug/kg	50	150	20
4,6-Dinitro-2-methylphenol	1600	ug/kg	26.63	ug/kg	20020206	1667	ug/kg	50	150	15	1667	ug/kg	50	150	20
2,4-Dinitrophenol	1600	ug/kg	53.581	ug/kg	19981104	1667	ug/kg	50	150	15	1667	ug/kg	50	150	20
2,4-Dinitrotoluene	330	ug/kg	72.59	ug/kg	20020206	1667	ug/kg	11	103	75	1667	ug/kg	10	123	75
2,6-Dinitrotoluene	330	ug/kg	61.50	ug/kg	20020206	1667	ug/kg	50	150	15	1667	ug/kg	50	150	20
Di-n-octyl phthalate	330	ug/kg	69.24	ug/kg	20020206	1667	ug/kg	50	150	15	1667	ug/kg	50	150	20
Fluoranthene	330	ug/kg	87.55	ug/kg	20020206	1667	ug/kg	50	150	15	1667	ug/kg	50	150	20

Target Analyte List: STL: SW-846 8270C

Matrix: SOLID
Extraction: SONICATION - Low Level
Method: Base/Neutrals and Acids (8270C)
QC Program: STANDARD TEST SET
Location: STL St. Louis

Target List 6010		Detection Limits				Check List 6151					Spike List 6152				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
Fluorene	330	ug/kg	87.64	ug/kg	20020206	1667	ug/kg	50	150	15	1667	ug/kg	50	150	20
Hexachlorobenzene	330	ug/kg	105.32	ug/kg	20020206	1667	ug/kg	50	150	15	1667	ug/kg	50	150	20
Hexachlorobutadiene	330	ug/kg	62.55	ug/kg	20020206	1667	ug/kg	50	150	15	1667	ug/kg	50	150	20
Hexachlorocyclopentadiene	1600	ug/kg	22.61	ug/kg	20020206	1667	ug/kg	50	150	15	1667	ug/kg	50	150	20
Hexachloroethane	330	ug/kg	28.621	ug/kg	19981104	1667	ug/kg	50	150	15	1667	ug/kg	50	150	20
Indeno(1,2,3-cd)pyrene	330	ug/kg	79.12	ug/kg	20020206	1667	ug/kg	50	150	15	1667	ug/kg	50	150	20
Isophorone	330	ug/kg	75.59	ug/kg	20020206	1667	ug/kg	50	150	15	1667	ug/kg	50	150	20
2-Methylnaphthalene	330	ug/kg	74.89	ug/kg	20020206	1667	ug/kg	50	150	15	1667	ug/kg	50	150	20
2-Methylphenol	330	ug/kg	61.88	ug/kg	20020206	1667	ug/kg	50	150	15	1667	ug/kg	50	150	20
4-Methylphenol	660	ug/kg	50.563	ug/kg	19981104	1667	ug/kg	50	150	15	1667	ug/kg	50	150	20
Naphthalene	330	ug/kg	62.15	ug/kg	20020206	1667	ug/kg	50	150	15	1667	ug/kg	50	150	20
2-Nitroaniline	1600	ug/kg	85.90	ug/kg	20020206	1667	ug/kg	50	150	15	1667	ug/kg	50	150	20
3-Nitroaniline	1600	ug/kg	98.27	ug/kg	20020206	1667	ug/kg	50	150	15	1667	ug/kg	50	150	20
4-Nitroaniline	1600	ug/kg	88.98	ug/kg	20020206	1667	ug/kg	50	150	15	1667	ug/kg	50	150	20
Nitrobenzene	330	ug/kg	57.98	ug/kg	20020206	1667	ug/kg	50	150	15	1667	ug/kg	50	150	20
2-Nitrophenol	330	ug/kg	45.91	ug/kg	20020206	1667	ug/kg	50	150	15	1667	ug/kg	50	150	20
4-Nitrophenol	1600	ug/kg	68.96	ug/kg	20020206	1667	ug/kg	20	100	75	1667	ug/kg	10	106	75
N-Nitrosodiphenylamine	330	ug/kg	95.21	ug/kg	20020206	1667	ug/kg	50	150	15	1667	ug/kg	50	150	20
N-Nitrosodi-n-propylamine	330	ug/kg	69.91	ug/kg	20020206	1667	ug/kg	10	112	75	1667	ug/kg	10	123	75
2,2'-oxybis(1-Chloropropane)	330	ug/kg	63.93	ug/kg	20020206	1667	ug/kg	50	150	15	1667	ug/kg	50	150	20
Pentachlorophenol	1600	ug/kg	61.04	ug/kg	20020206	1667	ug/kg	10	84	75	1667	ug/kg	10	90	75
Phenanthrene	330	ug/kg	98.53	ug/kg	20020206	1667	ug/kg	50	150	15	1667	ug/kg	50	150	20
Phenol	330	ug/kg	72.92	ug/kg	20020206	1667	ug/kg	10	106	75	1667	ug/kg	10	106	75
Pyrene	330	ug/kg	107.57	ug/kg	20020206	1667	ug/kg	24	105	75	1667	ug/kg	10	150	75
1,2,4-Trichlorobenzene	330	ug/kg	59.81	ug/kg	20020206	1667	ug/kg	10	106	75	1667	ug/kg	10	102	75
2,4,5-Trichlorophenol	330	ug/kg	92.82	ug/kg	20020206	1667	ug/kg	50	150	15	1667	ug/kg	50	150	20
2,4,6-Trichlorophenol	330	ug/kg	87.15	ug/kg	20020206	1667	ug/kg	50	150	15	1667	ug/kg	50	150	20
2-Fluorobiphenyl						1667	ug/kg	10	112	0	1667	ug/kg	10	116	0
2-Fluorophenol						1667	ug/kg	10	120	0	1667	ug/kg	10	112	0
2,4,6-Tribromophenol						1667	ug/kg	10	108	0	1667	ug/kg	10	117	0
Nitrobenzene-d5						1667	ug/kg	10	110	0	1667	ug/kg	10	116	0
Phenol-d5						1667	ug/kg	10	114	0	1667	ug/kg	10	113	0
Terphenyl-d14						1667	ug/kg	14	108	0	1667	ug/kg	10	137	0

Attachment QAPP-B4

Volatile Organics, GC/MS (8260B)

STL Reference Data Summary

Target Analyte List: STL: SW-846 8260B

Matrix: SOLID
Extraction: PURGE AND TRAP - 5 mL purge
Method: Volatile Organics, GC/MS (8260B)
QC Program: STANDARD TEST SET
Location: STL St. Louis

Target List 6051		Detection Limits				Check List 6147					Spike List 6148				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
Acetone	20	ug/kg	2.38	ug/kg	20020204	50	ug/kg	40	150	20	50	ug/kg	10	150	30
Benzene	5	ug/kg	0.14	ug/kg	20020204	50	ug/kg	78	117	20	50	ug/kg	80	119	30
Bromodichloromethane	5	ug/kg	0.14	ug/kg	20020204	50	ug/kg	81	120	20	50	ug/kg	76	118	30
Bromoform	5	ug/kg	0.25	ug/kg	20020204	50	ug/kg	74	124	20	50	ug/kg	66	123	30
Bromomethane	10	ug/kg	1.04	ug/kg	20020204	50	ug/kg	40	150	20	50	ug/kg	40	150	30
2-Butanone	20	ug/kg	1.68	ug/kg	20020204	50	ug/kg	35	150	20	50	ug/kg	35	150	30
Carbon disulfide	5	ug/kg	0.32	ug/kg	20020204	50	ug/kg	55	150	20	50	ug/kg	42	150	20
Carbon tetrachloride	5	ug/kg	0.24	ug/kg	20020204	50	ug/kg	77	118	20	50	ug/kg	67	128	30
Chlorobenzene	5	ug/kg	0.27	ug/kg	20020204	50	ug/kg	83	116	20	50	ug/kg	84	118	30
Dibromochloromethane	5	ug/kg	0.34	ug/kg	20020204	50	ug/kg	74	121	20	50	ug/kg	74	123	30
Chloroethane	10	ug/kg	0.54	ug/kg	20020204	50	ug/kg	55	150	20	50	ug/kg	54	150	20
Chloroform	5	ug/kg	0.23	ug/kg	20020204	50	ug/kg	78	126	20	50	ug/kg	86	123	30
Chloromethane	10	ug/kg	0.95	ug/kg	20020204	50	ug/kg	40	170	20	50	ug/kg	40	170	30
1,2-Dichlorobenzene	5	ug/kg	0.48	ug/kg	20020204	50	ug/kg	81	122	20	50	ug/kg	78	122	30
1,3-Dichlorobenzene	5	ug/kg	0.27	ug/kg	20020204	50	ug/kg	85	115	20	50	ug/kg	83	119	30
1,4-Dichlorobenzene	5	ug/kg	0.27	ug/kg	20020204	50	ug/kg	83	116	20	50	ug/kg	85	117	30
1,1-Dichloroethane	5	ug/kg	0.27	ug/kg	20020204	50	ug/kg	65	128	20	50	ug/kg	73	129	30
1,2-Dichloroethane	5	ug/kg	0.26	ug/kg	20020204	50	ug/kg	71	121	20	50	ug/kg	66	124	20
1,1-Dichloroethene	5	ug/kg	0.31	ug/kg	20020204	50	ug/kg	72	138	20	50	ug/kg	69	148	20
1,2-Dichloroethene (total)	10	ug/kg	0.55	ug/kg	20020204	100	ug/kg	80	119	20	100	ug/kg	78	124	30
1,2-Dichloropropane	5	ug/kg	0.22	ug/kg	20020204	50	ug/kg	65	127	20	50	ug/kg	72	122	30
cis-1,3-Dichloropropene	5	ug/kg	0.44	ug/kg	20020204	50	ug/kg	71	121	20	50	ug/kg	64	118	30
trans-1,3-Dichloropropene	5	ug/kg	0.80	ug/kg	20020204	50	ug/kg	58	134	20	50	ug/kg	62	133	30
Ethylbenzene	5	ug/kg	0.74	ug/kg	20020204	50	ug/kg	82	116	20	50	ug/kg	77	124	30
2-Hexanone	20	ug/kg	1.74	ug/kg	20020204	50	ug/kg	35	150	20	50	ug/kg	22	150	30
Methylene chloride	5	ug/kg	0.24	ug/kg	20020204	50	ug/kg	50	125	20	50	ug/kg	48	122	30
4-Methyl-2-pentanone	20	ug/kg	0.70	ug/kg	20020204	50	ug/kg	33	124	20	50	ug/kg	30	128	30
Styrene	5	ug/kg	0.36	ug/kg	20020204	50	ug/kg	80	119	20	50	ug/kg	72	121	30
1,1,2,2-Tetrachloroethane	5	ug/kg	0.42	ug/kg	20020204	50	ug/L	58	134	20	50	ug/kg	35	146	30
Tetrachloroethene	5	ug/kg	1.04	ug/kg	20020204	50	ug/kg	62	115	20	50	ug/kg	65	115	30
Toluene	5	ug/kg	0.24	ug/kg	20020204	50	ug/kg	79	115	20	50	ug/kg	69	133	30
1,1,1-Trichloroethane	5	ug/kg	0.28	ug/kg	20020204	50	ug/kg	71	126	20	50	ug/kg	77	128	30
1,1,2-Trichloroethane	5	ug/kg	0.63	ug/kg	20020204	50	ug/kg	67	120	20	50	ug/kg	65	130	30
Trichloroethene	5	ug/kg	0.22	ug/kg	20020204	50	ug/kg	76	115	20	50	ug/kg	68	127	30
Vinyl chloride	5	ug/kg	0.80	ug/kg	20020204	50	ug/kg	40	170	20	50	ug/kg	40	170	30
Xylenes (total)	10	ug/kg	0.97	ug/kg	20020204										
4-Bromofluorobenzene						50	ug/kg	73	126	0	50	ug/kg	76	143	0

Target Analyte List: STL: SW-846 8260B	Matrix: SOLID
	Extraction: PURGE AND TRAP - 5 mL purge
	Method: Volatile Organics, GC/MS (8260B)
	QC Program: STANDARD TEST SET
	Location: STL St. Louis

Target List 6051		Detection Limits				Check List 6147					Spike List 6148				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
1,2-Dichloroethane-d4						50	ug/kg	79	126	0	50	ug/kg	69	133	0
Toluene-d8						50	ug/kg	84	116	0	50	ug/kg	85	125	0
Dibromofluoromethane						50	ug/kg	85	115	0	50	ug/kg	78	119	0

Attachment QAPP-B5

Hydrocarbons, Volatile Petroleum (8015 MOD)

STL Reference Data Summary

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: PURGE AND TRAP - 5 mL purge
Method: Hydrocarbons, Volatile Petroleum (8015 MOD)
QC Program: STANDARD TEST SET
Location: STL St. Louis

Analyte List		Detection Limits				Check List 6019					Spike List 6020				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
Volatile Petroleum Hydrocarbons	0.1	mg/kg	0.05	mg/kg	20010804	1.0	mg/kg	58	139	14	1.0	mg/kg	30	158	50
Trifluorotoluene						0.02	mg/kg	65	131	0	0.02	mg/kg	50	150	0

Attachment QAPP-B6

Hydrocarbons, Extractable Petroleum (8015 MOD)

STL Reference Data Summary

Target Analyte List: STL: TPH Extractables 8015B Mod

Matrix: SOLID
Extraction: SONICATION - Low Level
Method: Hydrocarbons, Extractable Petroleum (8015 MOD)
QC Program: STANDARD TEST SET
Location: STL St. Louis

Target List 6028		Detection Limits				Check List 6036					Spike List 6037				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
Kerosene	25	mg/kg	25	mg/kg	20011018										
TPH (as Diesel)	25	mg/kg	1.36	mg/kg	20011018	83.3	mg/kg	39	113	63	83.3	mg/kg	30	170	25
o-Terphenyl						2.5	mg/kg	42	157	0	2.5	mg/kg	30	170	0

Attachment QAPP-B7

Inductively Coupled Plasma (6010B)

STL Reference Data Summary

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: TCLP(1311) -> METALS, TOTAL
Method: Inductively Coupled Plasma (6010B)
QC Program: STANDARD TEST SET
Location: STL St. Louis

Analyte List		Detection Limits				Check List 6038					Spike List 6039				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
Aluminum	1000	ug/L	20.3	ug/L	20020118	2500	ug/L	80	120	20	2500	ug/L	75	125	20
Antimony	100	ug/L	16.0	ug/L	20020118	2500	ug/L	80	120	20	2500	ug/L	75	125	20
Arsenic	200	ug/L	48.7	ug/L	19990723	12500	ug/L	80	120	20	12500	ug/L	75	125	20
Barium	200	ug/L	6.5	ug/L	20020118	12500	ug/L	80	120	20	12500	ug/L	75	125	20
Beryllium	50	ug/L	0.17	ug/L	20020118	2500	ug/L	80	120	20	2500	ug/L	75	125	20
Bismuth	500	ug/L	15.9	ug/L	20020118	1250	ug/L	80	120	20	5000	ug/L	75	125	20
Boron	500	ug/L	7.2	ug/L	20020118	1250	ug/L	80	120	20	5000	ug/L	75	125	20
Cadmium	50	ug/L	2.0	ug/L	20020118	2500	ug/L	80	120	20	2500	ug/L	75	125	20
Calcium	5000	ug/L	148	ug/L	20020118	125000	ug/L	80	120	20	50000	ug/L	75	125	20
Chromium	100	ug/L	2.0	ug/L	20020118	2500	ug/L	80	120	20	12500	ug/L	75	125	20
Cobalt	500	ug/L	4.0	ug/L	20020118	2500	ug/L	80	120	20	2500	ug/L	75	125	20
Copper	250	ug/L	7.7	ug/L	20020118	2500	ug/L	80	120	20	2500	ug/L	75	125	20
Iron	500	ug/L	14.5	ug/L	20020118	2500	ug/L	80	120	20	2500	ug/L	75	125	20
Lead	100	ug/L	9.0	ug/L	20020118	2500	ug/L	80	120	20	12500	ug/L	75	125	20
Lithium	500	ug/L	6.4	ug/L	20020118	625	ug/L	80	120	20	2500	ug/L	75	125	20
Magnesium	5000	ug/L	138	ug/L	20020118	125000	ug/L	80	120	20	12500	ug/L	75	125	20
Manganese	75	ug/L	0.79	ug/L	20020118	2500	ug/L	80	120	20	2500	ug/L	75	125	20
Molybdenum	400	ug/L	7.1	ug/L	20020118	625	ug/L	80	120	20	2500	ug/L	75	125	20
Nickel	400	ug/L	12.0	ug/L	20020118	2500	ug/L	80	120	20	2500	ug/L	75	125	20
Potassium	5000	ug/L	1330	ug/L	20020118	125000	ug/L	80	120	20	12500	ug/L	75	125	20
Selenium	200	ug/L	45.6	ug/L	19990723	2500	ug/L	80	120	20	2500	ug/L	75	125	20
Silicon	500	ug/L	29.1	ug/L	20020118	1250	ug/L	80	120	20	5000	ug/L	75	125	20
Silver	100	ug/L	6.0	ug/L	20020118	625	ug/L	49.2	134.	20	2500	ug/L	49.2	134.	20
Sodium	5000	ug/L	140	ug/L	20020118	125000	ug/L	80	120	20	12500	ug/L	75	125	20
Strontium	250	ug/L	1.5	ug/L	20020118	625	ug/L	80	120	20	2500	ug/L	75	125	20
Tellurium	500	ug/L	61.9	ug/L	19990723	1250	ug/L	80	120	20	5000	ug/L	75	125	20
Thallium	200	ug/L	51.2	ug/L	19990723	5000	ug/L	80	120	20	2500	ug/L	75	125	20
Thorium	500	ug/L	32.3	ug/L	20020118	1250	ug/L	80	120	20	5000	ug/L	75	125	20
Tin	500	ug/L	19.8	ug/L	20020118	1250	ug/L	80	120	20	5000	ug/L	75	125	20
Uranium	500	ug/L	186	ug/L	20020118	1250	ug/L	80	120	20	5000	ug/L	75	125	20
Vanadium	500	ug/L	2.3	ug/L	20020118	2500	ug/L	80	120	20	2500	ug/L	75	125	20
Zinc	200	ug/L	1.3	ug/L	20020118	2500	ug/L	80	120	20	2500	ug/L	75	125	20

Attachment QAPP-B8

Inductively Coupled Plasma "ICP" (CLP)

STL Reference Data Summary

Target Analyte List: STL: CLP90 and SW846 Metals ICAP (22)

Matrix: SOLID
Extraction: METALS, TOTAL - Soils
Method: Inductively Coupled Plasma "ICP" (CLP)
QC Program: CLP: ILMO3.0 AND OLMO1.8
Location: STL St. Louis

Target List 6007		Detection Limits				Check List 6005					Spike List 6013				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
Aluminum	40	mg/kg	10.82	mg/kg	20020201	9200	mg/kg	58	142	20	200	mg/kg	75	125	20
Antimony	12	mg/kg	7.62	mg/kg	20020201	62.7	mg/kg	27	225	20	50	mg/kg	75	125	20
Barium	40	mg/kg	0.54	mg/kg	20020201	509	mg/kg	77	123	20	200	mg/kg	75	125	20
Beryllium	1	mg/kg	0.04	mg/kg	20020201	55.9	mg/kg	78	122	20	5	mg/kg	75	125	20
Cadmium	1	mg/kg	0.50	mg/kg	20020201	157	mg/kg	75	125	20	5	mg/kg	75	125	20
Calcium	1000	mg/kg	18.68	mg/kg	20020201	11700	mg/kg	75	125	20	5000	mg/kg	75	125	20
Chromium	2	mg/kg	0.76	mg/kg	20020201	51.4	mg/kg	76	124	20	20	mg/kg	75	125	20
Cobalt	10	mg/kg	0.56	mg/kg	20020201	88.4	mg/kg	78	122	20	50	mg/kg	75	125	20
Copper	5	mg/kg	1.08	mg/kg	20020201	69.5	mg/kg	82	118	20	25	mg/kg	75	125	20
Iron	20	mg/kg	5.38	mg/kg	20020201	13700	mg/kg	61	139	20	100	mg/kg	75	125	20
Magnesium	1000	mg/kg	30.2	mg/kg	20020201	3070	mg/kg	74	126	20	5000	mg/kg	75	125	20
Manganese	3	mg/kg	0.24	mg/kg	20020201	674	mg/kg	76	124	20	50	mg/kg	75	125	20
Nickel	8	mg/kg	2.0	mg/kg	20020201	112	mg/kg	78	122	20	50	mg/kg	75	125	20
Potassium	1000	mg/kg	368	mg/kg	20020201	3640	mg/kg	73	127	20	5000	mg/kg	75	125	20
Silver	2	mg/kg	1.40	mg/kg	20020201	84.3	mg/kg	64	135	20	5	mg/kg	75	125	20
Sodium	1000	mg/kg	25.0	mg/kg	20020201	863	mg/kg	68	132	20	5000	mg/kg	75	125	20
Vanadium	10	mg/kg	1.20	mg/kg	20020201	136	mg/kg	68	132	20	50	mg/kg	75	125	20
Zinc	4	mg/kg	1.48	mg/kg	20020201	289	mg/kg	78	123	20	50	mg/kg	75	125	20

Attachment QAPP-B9

Pesticides (8081A)

STL Reference Data Summary

Target Analyte List: STL: Pesticides by 8081A

Matrix: SOLID
Extraction: SONICATION - Low Level
Method: Pesticides (8081A)
QC Program: STANDARD TEST SET
Location: STL St. Louis

Target List 6002		Detection Limits				Check List 6012					Spike List 6011				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
Aldrin	1.7	ug/kg	0.21	ug/kg	20020116	16.7	ug/kg	57	150	20	16.7	ug/kg	70	115	30
alpha-BHC	1.7	ug/kg	0.38	ug/kg	20020116	16.7	ug/kg	56	150	20	16.7	ug/kg	72	115	30
beta-BHC	1.7	ug/kg	0.30	ug/kg	20020116	16.7	ug/kg	53	150	20	16.7	ug/kg	29	150	30
delta-BHC	1.7	ug/kg	0.27	ug/kg	20020116	16.7	ug/kg	49	141	20	16.7	ug/kg	62	115	30
gamma-BHC (Lindane)	1.7	ug/kg	0.27	ug/kg	20020116	16.7	ug/kg	59	150	20	16.7	ug/kg	80	117	30
4,4'-DDD	1.7	ug/kg	0.79	ug/kg	20020116	16.7	ug/kg	60	149	20	16.7	ug/kg	59	149	30
4,4'-DDE	1.7	ug/kg	1.16	ug/kg	20020116	16.7	ug/kg	65	150	20	16.7	ug/kg	73	123	30
4,4'-DDT	1.7	ug/kg	0.97	ug/kg	20020116	16.7	ug/kg	66	150	20	16.7	ug/kg	66	150	30
Dieldrin	1.7	ug/kg	1.07	ug/kg	20020116	16.7	ug/kg	57	150	20	16.7	ug/kg	62	145	30
Endosulfan I	1.7	ug/kg	0.43	ug/kg	20020116	16.7	ug/kg	60	146	20	16.7	ug/kg	73	118	30
Endosulfan II	1.7	ug/kg	0.79	ug/kg	20020116	16.7	ug/kg	59	150	20	16.7	ug/kg	78	115	30
Endosulfan sulfate	1.7	ug/kg	0.66	ug/kg	20020116	16.7	ug/kg	59	148	20	16.7	ug/kg	75	115	30
Endrin	1.7	ug/kg	0.41	ug/kg	20020116	16.7	ug/kg	62	150	20	16.7	ug/kg	85	125	30
Heptachlor	1.7	ug/kg	0.42	ug/kg	20020116	16.7	ug/kg	58	150	20	16.7	ug/kg	52	150	30
Heptachlor epoxide	1.7	ug/kg	0.90	ug/kg	20020116	16.7	ug/kg	62	150	20	16.7	ug/kg	73	124	30
Toxaphene	67	ug/kg	42	ug/kg	19990819										
Decachlorobiphenyl						6.67	ug/kg	59	146	0	6.67	ug/kg	45	147	0
Tetrachloro-m-xylene						6.67	ug/kg	66	133	0	6.67	ug/kg	57	116	0

Attachment QAPP-B10

PCBs (8082)

STL Reference Data Summary

Target Analyte List: STL: Aroclors

Matrix: SOLID
Extraction: SONICATION w/ACID STRIP (PCB)
Method: PCBs (8082)
QC Program: STANDARD TEST SET
Location: STL St. Louis

Target List 6008		Detection Limits				Check List 6192					Spike List 6193				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
Aroclor 1016	33	ug/kg	12.14	ug/kg	20020115	167	ug/kg	70	141	15	167	ug/kg	50	150	64
Aroclor 1221	33	ug/kg	12.14	ug/kg	20020115										
Aroclor 1232	33	ug/kg	12.14	ug/kg	20020115										
Aroclor 1242	33	ug/kg	12.14	ug/kg	20020115										
Aroclor 1248	33	ug/kg	12.14	ug/kg	20020115										
Aroclor 1254	33	ug/kg	7.13	ug/kg	20020115										
Aroclor 1260	33	ug/kg	7.13	ug/kg	20020115	167	ug/kg	73	139	15	167	ug/kg	50	150	64
Aroclor 1262	33	ug/kg	7.13	ug/kg	20020115										
Aroclor 1268	33	ug/kg	7.13	ug/kg	20020115										
Decachlorobiphenyl						167	ug/kg	59	150	0	167	ug/kg	50	150	0

ENTACT
Appendix
C

ATTACHMENT QAPP C

SEVERN TRENT LABORATORY STANDARD OPERATING PROCEDURES

Attachment QAPP-C1

Inductively Coupled Plasma-Atomic Emission Spectroscopy
Method 200.7 CLP- M, SOW ILMO3.0

SOP No.:	CORP-MT-0002STL
Revision No.:	2.2
Revision Date:	11/03/00
Page:	1 of 39
Implementation Date:	04/24/01

SEVERN TRENT SERVICES

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STL ST. LOUIS STANDARD OPERATING PROCEDURE

TITLE: INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION SPECTROSCOPY, METHOD 200.7 CLP-M, SOW ILMO3.0

(SUPERSEDES: REVISION 2.1)

Prepared by: _____

Approved by: _____
Technical Specialist

Approved by: _____
Quality Assurance Manager

Approved by: _____
Environmental Health and Safety Coordinator

Approved by: _____
Laboratory Director

1. SCOPE AND APPLICATION

1.1. This procedure describes the analysis of certain metals by Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP) and is applicable to the determination of dissolved, suspended, total recoverable and total elements in ground water, aqueous samples, soils, sludges, wastes, sediments, TCLP, EP and other leachates/extracts. All matrices require digestion prior to analysis with the exception of analyses for dissolved metals in filtered and acidified aqueous samples. Although digestion is not specifically required by the method, some clients and regulators do require digestion of dissolved samples and this must be clarified before project initiation.

1.2. This SOP is based on Method 200.7 CLP-M as contained in SOW ILMO3.0.

1.3. In addition to SOW ILMO3.0, this SOP is also compliant with the requirements of CLP SOWs 7/88, 3/90, ILMO1.0 and ILMO2.1.

1.4. ICP analysis provides for the determination of metal concentrations over several orders of magnitude. Detection limits, sensitivity and optimum concentration ranges of the metals will vary with the matrices and instrumentation used.

1.5. Table I of Appendix A lists the elements approved for analysis by CLP SOW ILMO3.0. Table II of Appendix A lists additional elements that may be analyzed under this SOP provided the method performance criteria presented in Section 16.0 are met. Reporting limits will be proportionately higher for sample extracts that require dilution and for soil samples that require concentration adjustments to account for % moisture.

1.6. Method detection limits are maintained in the Information Management System (QuantIMS). Because of their dynamic nature, they are not specifically listed in this document, but can be retrieved at any time using TraQAr tools.

1.7. Quality control limits (accuracy and precision for spikes) are also maintained in QuantIMS, and are also dynamic. Therefore, they are not specifically listed in this document, but can be retrieved at any time using TraQAr tools.

1.8. Additional elements may be amenable to this method. The minimum requirement for non-standard analytes is that the reporting limit be set at the lowest required concentration that can actually be detected by the instrument, and when an MDL study can not be conducted, the MDL be set equal to the reporting limit.

2. SUMMARY OF METHOD

2.1. This method describes a technique for the determination of elements in solution. The basis of the method is the measurement of atomic emission by an optical spectroscopic technique. Samples are nebulized and the aerosol that is produced is transported to the plasma torch where excitation occurs. Characteristic atomic-line emission spectra are produced by a radio frequency inductively coupled plasma (ICP). The spectra are dispersed by a grating spectrometer and the intensities of the lines are monitored by photomultiplier tubes. The photocurrents from the photomultiplier tubes are processed and controlled by a computer system. A background correction technique is required to compensate for variable background contribution to the determination of trace elements. Background must be measured adjacent to analyte lines during analysis. The position selected for the background intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. The position used must be free of spectral interferences and reflect the same change in background intensity as occurs at the analyte wavelength measured. Background correction is not required in cases where a background correction measurement would actually degrade the analytical result. The possibility of additional interferences should also be recognized and appropriate actions taken.

2.2. Refer to the appropriate SOPs for details on sample preparation methods.

3. DEFINITIONS

3.1. See policy QA-003 for definitions

3.2. Dissolved Metals: Those elements that will pass through a 0.45 um membrane filter. (Sample is acidified after filtration)

3.3. Suspended Metals: Those elements that are retained by a 0.45 um filter.

3.4. Total Metals: The concentration determined on an unfiltered sample following vigorous digestion.

3.5. Total Recoverable Metals: The concentration determined on an unfiltered sample following treatment with hot, dilute mineral acid.

4. INTERFERENCES

4.1. Spectral, physical and chemical interference effects may contribute to inaccuracies in the determinations of trace elements by ICP. Spectral interferences are caused by:

- Overlap of a spectral line from another element.
- Unresolved overlap of molecular band spectra.
- Background contribution from continuous or recombination phenomena.
- Stray light from the line emission of high concentration elements.

4.1.1. A background correction technique is required to compensate for variable background contribution to the determination of trace elements. Background correction is not required in cases where a background corrective measurement would actually degrade the analytical result.

4.1.2. Inter-element correction factors (IECs) are necessary to compensate for spectral overlap. Inter-element interferences occur when elements in the sample emit radiation at wavelengths so close to that of the analyte that they contribute significant intensity to the analyte channel. If such conditions exist, the intensity contributed by the matrix elements will cause an excessively high (or sometimes low) concentration to be reported for the analyte. Inter-element corrections (IECs) must be applied to the analyte to remove the effects of these unwanted emissions. To calculate an IEC, divide the observed concentration of the analyte by the observed concentration of the "interfering element."

4.1.3. Physical interferences are generally considered to be effects associated with sample transport, nebulization and conversion within the plasma. These interferences may result in differences between instrument responses for the sample and the calibration standards. Physical interferences may occur in the transfer of solution to the nebulizer (e.g., viscosity effects), at the point of aerosol formation and transport to the plasma (e.g., surface tension) or during excitation and ionization processes within the plasma itself. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If physical interferences are present, dilution of the sample, use of a peristaltic pump, use of an internal standard and/or use of a high solids nebulizer can reduce the effect.

4.1.4. Chemical interferences are characterized by molecular compound formation, ionization effects and solute vaporization effects. Normally these effects are not significant with the ICP technique but if observed can be minimized by buffering the sample, matrix matching or standard addition procedures.

5. SAFETY

5.1. Procedures shall be carried out in a manner that protects the health and safety of all associates.

5.2. The Chemical Hygiene Plan (CHP) gives details about the specific health and safety practices that are to be followed in the laboratory area. Personnel must receive training in the CHP, including the written Hazard Communication plan, prior to working in the laboratory. Consult the CHP, the Health and Safety Policies and

Procedures Manual, and available Material Safety Data Sheets (MSDS) prior to using the chemicals in the method.

5.3. Consult the Health and Safety Policies and Procedures Manual for information on Personal Protective Equipment. Eye protection that satisfies ANSI Z87.1 (as per the Chemical Hygiene Plan), laboratory coat and appropriate gloves must be worn while samples, standards, solvents and reagents are being handled. Disposable gloves that have become contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.4. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the Material Safety Data Sheets (MSDS) files maintained in the laboratory.

5.4.1. The following materials are known to be **corrosive**:
Sulfuric acid, hydrochloric acid, nitric acid and hydrofluoric acid. (NOTE: sulfuric and hydrofluoric acids are used in cleaning the ICP torch and hydrofluoric acid is also commonly used in air toxics preparations.)

5.4.2. The following materials are known to be **oxidizing agents:**
Nitric acid and hydrogen peroxide

5.4.3. The plasma emits strong UV light and is harmful to vision. AVOID looking directly at the plasma.

5.4.4. The RF generator produces strong radio frequency waves, most of which are unshielded. People with pacemakers should not go near the instrument while in operation.

5.5. Exposure to chemicals will be maintained **as low as reasonably achievable,** therefore, unless they are known to be non-hazardous, all samples will be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Metal digestates can be processed outside of a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.

5.6. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operations will permit or under other means of mechanical ventilation.

5.7. All work must be stopped in the event of a known, or potential compromise to the health or safety of any associate. The situation must be reported **immediately** to a laboratory supervisor.

5.8. The use of hydrofluoric acid requires special safety precautions. Consult the facility EH&S Manager and laboratory supervisor for guidance.

5.9. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices outlined in the Health and Safety Manual. These employees must have training on the hazardous waste disposal practices initially upon assignment of these tasks, followed by an annual refresher training.

6. EQUIPMENT AND SUPPLIES

6.1. Inductively Coupled Plasma Atomic Emission Spectrometer equipped with autosampler and background correction.

6.2. Radio Frequency Generator

6.3. Argon gas supply, welding grade or equivalent

6.4. Coolflow or appropriate water cooling device

6.5. Peristaltic Pump

6.6. Calibrated automatic pipettes or Class A glass volumetric pipettes

6.7. Class A volumetric flasks

6.8. Autosampler tubes

7. REAGENTS AND STANDARDS

7.1. Reagents

7.1.1. All reagent preparation is documented in the electronic standards log. All reagents are labeled with their unique ID, name (and concentration, if applicable) of the reagent, the date prepared and the expiration date.

7.1.2. Concentrated nitric acid (HNO3), trace metal grade or better

7.1.3. Concentrated hydrochloric acid (HCl), trace metal grade or better

7.1.4. Reagent water must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of preparation blanks.

7.2. Standards

7.2.1. All standards preparation, documentation and labeling must follow the requirements of STL-QA-0002.

7.2.2. Intermediate standards are purchased as custom multi-element mixes or as single-element solutions. All standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. Intermediate standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the intermediate solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem.

7.2.3. Working calibration and calibration verification solutions may be used for up to 1 month and must be replaced sooner if verification from an independent source indicates a problem. Standards should be prepared in a matrix of 5% hydrochloric and 5% nitric acids.

7.2.4. Refer to Tables III, IV, V and VI (Appendix A) for details regarding the working standard concentrations for calibration, calibration verification, interference correction and spiking solutions.

7.2.5. For analyses of solid and soil matrices a solid reference material is obtained commercially for the laboratory control standard.

7.2.6. Initial calibration verification (ICV) standards are similar to calibration standards, but are from a completely different source.

7.2.6.1. For analyses performed under direct contract to the USEPA CLP, EPA provided ICV and ICS solutions must be used in place of the custom standards.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Sample holding times for metals are 180 days from time of receipt to the time of analysis.

8.2. Aqueous samples are preserved with nitric acid to a pH of <2 and may be stored in either plastic or glass. Refrigeration is not required. If boron or silica are to be determined, plastic containers are preferred.

8.3. Soil samples do not require preservation but must be stored at 4°C ± 2° until the time of analysis.

9. QUALITY CONTROL

9.1. QC requirements

9.1.1. Each analytical batch may contain up to 20 environmental samples, a method blank, and a single Laboratory Control Sample (LCS) and an MS/MSD pair. In the event that there is not sufficient sample to analyze an MS/MSD, an LCS duplicate (LCSD) is prepared and analyzed.

9.1.1.1. Samples that have assigned QC limits different than the standard limits contained in QuantIMS QC code 01 must be batched separately, but can share the same QC samples.

9.1.1.2. Additional MS/MSDs do not count towards the 20 samples in an analytical batch.

9.1.1.3. A method blank must be included with each batch of samples. The matrix for aqueous and soil analyses is reagent (DI) water.

9.1.1.4. The LCS is spiked with all of the standard target compounds and is used to monitor the accuracy of the analytical process, independent of matrix effects. The matrix for aqueous analyses is reagent water (DI) and a standard reference material for solids.

9.1.1.5. All LCS, MS/MSD and surrogate results -- whether they pass criteria or not -- are uploaded into 's QuantIMS system for maintenance and periodic update of limits.

9.1.2. Instrument conditions must be the same for all standards, samples and QC samples.

9.1.3. All data will be reviewed by the analyst (1st review level) and then by a peer or supervisor (2nd level review).

9.2. Documentation

The CLP SOW ILMO3.0 document provides further details of the QC and corrective action guidelines presented in this SOP. Refer to this document if additional guidance is required.

Table VI of Appendix A provides a summary of quality control requirements including type, frequency, acceptance criteria and corrective action.

9.2.1. Initial Demonstration of Capability

Prior to analysis of any analyte using Method 200.7 CLP-M the following requirements must be met.

9.2.1.1. Instrument Detection Limit (IDL) - The IDL for each analyte must be determined for each analyte wavelength used on each instrument. The IDL must be determined quarterly (every 3 months). If the instrument is adjusted in anyway that may affect the IDL, the IDL for that instrument must be redetermined. The IDL shall be determined by multiplying by 3, the average of the standard deviations obtained on three nonconsecutive days from the analysis of a standard solution (each analyte in reagent water) at a concentration 3x - 5x the previously determined IDL, with seven consecutive measurements per day. Each measurement must be performed as though it were a separate analytical sample (i.e., each measurement must be followed by a rinse and/or any other procedure performed between the analysis of separate samples).

- If the IDLs are not completed by the quarterly due date no CLP analyses may be performed until the IDLs are completed (IDLs can not be retroactively applied).
- For reporting purposes, if multiple ICP instruments are used for the analysis of an element in a SDG, the highest ICP IDL must be used for reporting concentration values.
- Instrument specific IDLs must be used for all calculations.

9.2.1.2. Linear Range Verification (LR) - The linear range must be determined on a quarterly basis for each analyte wavelength used on each instrument. The standards used to define the linear range limit must be analyzed during a routine analytical run. The determined concentration of the linear range standards must be within 5% of the true value. The linear range is the concentration above which results cannot be reported without dilution of the sample. If the instrument is adjusted in any way that may affect the LRs, the LRs must be redetermined.

9.2.1.3. Background Correction Points - To determine the appropriate location for off-line background correction when establishing methods, the user must scan the area on either side adjacent to the wavelength and record the apparent emission intensity from all other method analytes. This spectral information must be documented and

kept on file. Background correction points must be set prior to determining IECs. Refer to the ICP instrument manual for specific procedures to be used in setting background correction points.

9.2.1.4. Inter-element Corrections (IECs) - ICP interelement correction factors must be determined prior to the analysis of samples and annually thereafter. Correction factors for spectral interferences due to Al, Ca, Fe and Mg must be determined for all ICPs at all wavelengths used for reporting results. Correction factors for spectral interferences other than Al, Ca, Fe or Mg must be documented if applied. If the instrument is adjusted in any way that may affect the IECs, the IECs must be redetermined. When initially determining IECs for an instrument, wavelength scans must be performed to ensure that solutions in use are free from contaminants. If an IEC varies significantly from the previously determined IEC then the possibility of contamination should be investigated. The purity of the IEC check solution can be verified by using a standard from a second source or an alternate method (i.e., GFAA or ICP-MS). Published wavelength tables (e.g. MIT tables, Inductively Coupled Plasma-Atomic Spectroscopy: Prominent Lines) can also be consulted to evaluate the validity of the IECs. Refer to the ICP instrument manufacturer's recommendations for specific procedures to be used in setting IECs. An IEC must be established to compensate for any interelement interference which results in a false analyte signal greater than ± the RL as defined in Tables I, IA or II.

Note: Trace ICP IECs are more sensitive to small changes in the plasma and instrument setup conditions. Adjustments in the IECs will be required on a more frequent basis for the Trace as reflected by the ICSA response.

9.2.1.5. Rinse Time Determination - Rinse times must be determined prior to placing a new ICAP into service or when sample introduction systems are modified in such a way as to effect sample introduction rates. To determine the appropriate rinse time for a particular ICP system, a linear range verification standard (see 9.2.1.2) or other suitable high standard should be aspirated as a regular sample followed by the analysis of a series of rinse blanks. The length of time required to reduce the analyte signals to < RL will define the rinse time for a particular ICP system. For some analytes it may be impractical to set the rinse time based on the linear range standard result (i.e., analyte not typically detected in environmental samples at that level and an excessive rinse time would be required at the linear range level). Rinse time studies can be conducted at additional concentration levels. These additional studies must be documented and kept on file, if a concentration other than the linear range level is used to set the rinse time. The concentration levels used to establish the rinse time must be taken into consideration when reviewing the data.

9.2.2. Sample Delivery Group (SDG) - An SDG is defined in the CLP SOW by the following, whichever is more frequent:

- each Case of field samples received, OR
- each 20 field samples within a Case, OR
- each 14 day calendar day period during which field samples in a Case are received.

For commercial clients the SDG definition must be clarified with the client prior to the analysis of samples.

9.2.3. Preparation Batch - The CLP SOW defines batch as "A group of samples prepared at the same time". Preparation batches are also separated by matrix into one of the following types: aqueous or solid.

9.2.4. Analytical Sample - The CLP SOW defines the term "analytical sample" to include all field samples and all required QC samples (matrix spikes, duplicates, postdigestion spikes, serial dilutions, laboratory control samples, interference check samples, CRDL standards, preparation blanks and linear range analyses) except those directly related to instrument calibration or calibration verification (calibration standards, ICV/ICB and CCV/CCB).

9.2.5. Preparation Blank (PB) - One preparation blank (method blank) must be processed with each preparation batch. The preparation blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The preparation blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The preparation blank should not contain any analyte of interest at or above the contract required detection limit (exception: see below).

- If any analyte concentration in the blank is above the CRDL, the lowest concentration of that analyte in the associated samples must be 10X the blank concentration. Otherwise, all samples associated with the blank, with the analyte's concentration less than 10X the blank concentration and above the CRDL, must be redigested and reanalyzed for that analyte (except for identified aqueous soil field blanks).
- If the concentration of the blank is below the negative CRDL, then all samples reported below 10X CRDL associated with the blank must be redigested and reanalyzed.
- If the above criteria are not met and reanalysis is not possible, then the sample data must be qualified. **This anomaly must be addressed in the project narrative and the client must be notified.**

9.2.6. Laboratory Control Sample (LCS) - One LCS must be processed with each preparation batch or SDG whichever is more frequent. The LCS must be carried through the entire analytical procedure. The LCS is used to monitor the accuracy of the analytical process. An aqueous LCS must contain all analytes of interest and must be processed with each batch of aqueous samples. Aqueous LCS spiking levels are provided in Table III of Appendix A. A control limit of 80-120% recovery is applied to the aqueous LCS (exception: Ag and Sb). The solid LCS material must be processed with each batch of solid samples. The solid LCS control limits shall be those established by the supplier of the solid LCS. No control limits shall be applied to any analyte present in the soil LCS at a concentration less than the CRDL. Corrective action when LCS results fail to meet control limits will be repreparation and reanalysis of the batch.

9.2.7. Matrix Spike (MS)- One matrix spike must be processed for each SDG or each group of samples of a similar matrix type, whichever is more frequent. A matrix spike is a field sample to which known concentrations of target analytes have been added prior to digestion. Samples identified as field blanks cannot be used for matrix spike analysis. The matrix spike results are used to determine the effect of a matrix on the accuracy of the analytical process. Spiking levels are provided in Table III of Appendix A.

- If any analyte recovery falls outside the control limits of 75-125%, the data of all samples associated with that spiked sample must be flagged appropriately. An exception to the rule is granted if the native analyte concentration in the MS exceeds 4x the spike level for that analyte. In such an event the data shall be reported unflagged even if the recovery does not meet the criteria.
- The average of the duplicate results cannot be used for the purpose of determining percent recovery.
- If the MS recovery falls outside the control limits and the sample result does not exceed 4x the spike added, a post digestion spike must be performed for those elements that do not meet the specified criteria (exception: Ag).
- The post-digestion spike is performed by spiking the unspiked sample aliquot with two times the analyte level or two times the CRDL, whichever is greater.

- For dissolved metals samples which have not been digested, a MS must be performed per SDG by spiking the undigested sample with the spiking levels in Table III of Appendix A.

9.2.8. Duplicate (D)- One duplicate must be processed for each SDG or each group of samples of a similar matrix type, whichever is more frequent. Samples identified as field blanks cannot be used for duplicate sample analysis.

- If the sample results are greater than or equal to five times the CRDL, a control limit of ± 20% RPD must be used.
- If either the sample or sample duplicate value is less than five times the CRDL, a control limit of ± the CRDL must be used.
- If both the sample and sample duplicate results are below the IDL, the RPD is not calculated and no control criteria apply.
- If the duplicate RPD is outside control limits the associated data is flagged appropriately.

9.2.9. Serial Dilution (L) - Serial dilution analysis is performed to determine whether significant physical or chemical interferences exist due to the sample matrix. One sample per SDG or each group of samples of a similar matrix type, whichever is more frequent, must be processed as a serial dilution sample. The serial dilution is performed by running a sample at a 5x dilution. Samples identified as field blanks cannot be used for serial dilution analyses. The results of the diluted sample, after correction for the dilution, should agree within 10% of the original sample determination when the original sample concentration is greater than 50x the IDL. If the serial dilution analysis fails for an analyte a chemical or physical interference should be suspected and the data for the associated samples must be flagged appropriately.

9.2.10. Initial Calibration Verification (ICV/ICB) - Calibration accuracy is verified by analyzing a second source standard (ICV) immediately after calibration. The ICV result must fall within 10% of the true value for that solution. An ICB is analyzed immediately following the ICV to monitor low level accuracy and system cleanliness. The ICB result must fall within ± the CRDL from zero. If either the ICV or ICB fail to meet criteria, the analysis should be terminated, the problem corrected, the instrument recalibrated and the calibration reverified. (See Section 11.10 or 11.11 for required run sequence).

9.2.11. Continuing Calibration Verification (CCV/CCB) - Calibration accuracy is monitored throughout the analytical run through the analysis of a known standard after every 10 samples or every 2 hours whichever is more frequent. The CCV must be a midrange standard. The CCV result must fall within 10% of the true value for that solution. A CCB is analyzed immediately following each CCV. The CCB result must fall within ± CRDL from zero. (See Section 11.10 for required run sequence.) If either a CCV or CCB fail to meet criteria, the analysis for the affected element must be terminated, the instrument recalibrated, the calibration verified and all analytical samples analyzed since the last compliant CCV/CCB rerun. (See Section 11.11 for an illustration of the appropriate reanalysis sequence).

- Each CCV analyzed must reflect the conditions of analysis of all associated samples. The duration of analysis, rinses, and other operations that may affect the CCV result may not differ from those used for the associated samples. For example, the difference in time between a CCV analysis and the CCB immediately following it or the sample immediately preceding it may not exceed the smallest difference between any two consecutive samples associated with that CCV.

9.2.12. Interference Check Analysis (ICSA/ICSAB) - The validity of the interelement correction factors is demonstrated through the successful analysis of interference check solutions. The ICSA contains only interfering elements, the ICSAB contains analytes and interferents. Refer to Table V of Appendix A for the details of ICSA and ICSAB composition. All analytes must be spiked into the ICSAB solution, therefore, if a non-routine analyte is required then it must be manually spiked into the ICSAB using a certified ultra high purity single element solution or

custom lab-specific mix (exception: USEPA CLP contract analyses). Elements known to be interferents on a required analyte must be included in the ICP run when that analyte is determined. Aluminum, iron, calcium and magnesium must always be included in all ICP runs.

9.2.12.1. The ICSA and ICSAB solutions must be run at the beginning and end of the run or every 8 hours, whichever is more frequent. (See Section 11.10 or 11.11 for required run sequence.)

9.2.12.2. The ICSAB results for the interferents must fall within 80 - 120% of the true value. If any ICSAB interferent result fails criteria, the analysis should be terminated, the problem corrected and the instrument recalibrated and the samples rerun.

9.2.12.3. The ICSAB analytes must be recovered within 80 - 120% of the true value. If the ICSAB analytes do not meet criteria the analysis must be terminated, the problem corrected, the instrument recalibrated and the samples rerun for the affected analytes.

9.2.12.4. ICSA results for the non-interfering elements with reporting limits ≤ 10 ug/L must fall within the guidelines of +/- 2x CRDL from zero. If the ICSA results for the non-interfering elements do not fall within +/- 2x CRDL from zero the field sample data must be evaluated as follows:

- If the non-interfering element concentration in the ICSA is the result of contamination versus a spectral interference, and this reason is documented, the field sample data can be accepted.
- If the affected element was not required then the sample data can be accepted.
- If the interfering elements are not present in the field sample at a concentration which would result in a false positive or negative result greater than ± 2x CRDL from zero then the field sample data can be accepted.
- If the interfering element is present in the field sample at a level which would result in a false analyte signal greater than ± 2x CRDL from zero, the data can be accepted only if the concentration of the affected analyte in the field sample is more than 10x the analyte signal in the ICSA.
- If the data does not meet the above conditions then the IECs must be re-evaluated and corrected if necessary and the affected samples reanalyzed or the sample results manually corrected through application of the new IEC to the raw results. If the results are recalculated manually the calculations must be clearly documented on the raw data.

9.2.13. CRI - To verify linearity near the CRDL for ICP analysis, a CRI standard is run at the beginning and end of each sample analysis run, or a minimum of twice per 8 hour shift, whichever is more frequent, but not before the ICV. The CRI standard must contain the analytes at two times the CRDL or two times the IDL, whichever is greater. The CRI must be run for all analytes except Al, Ba, Ca, Fe, Mg, Na and K. (See Section 11.10 or 11.11 for required run sequence).

Note: The custom STL CRI mix contains all analytes at a level two times the CRDL including the elements excluded above as defined in Tables IV and IVA. Lab specific customization of the CRI will be required for As, Se, Pb and Tl for analyses conducted on the non-Trace ICP (i.e., 2x IDL).

9.2.14. Quality Assurance/Project Summaries - Certain clients may require specific project or program QC which may supersede the SOP requirements. Quality Assurance Summaries (QAS) or equivalent documents providing project specific requirements should be developed so that the project staff clearly understand the special project requirements.

9.3. Procedural Variations

9.3.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of the analyst to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation shall be completely documented using a

Nonconformance Memo and approved by the Supervisor and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

9.3.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

9.4. Nonconformance and Corrective Action

Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the facility QA Manager.

9.5. QC Program

Further details of QC and corrective action guidelines are presented in the QC Program policy document (QA-003).

10. CALIBRATION AND STANDARDIZATION

10.1. Initial Calibration

10.1.1. Set up the instrument with the operating parameters recommended by the manufacturer. Allow the instrument to become thermally stable before beginning calibration (approximately 30 minutes of warm-up is required).

10.1.2. Profile and calibrate the instrument according to the instrument manufacturer's instructions. Refer to the ICP instrument manual for a detailed set up and operation protocols.

10.1.3. Calibration must be performed daily or once every 24 hours and each time the instrument is set up. The instrument standardization date and time must be included in the raw data.

10.1.4. If the calibration curve does not meet method requirements,

10.1.4.1. Evaluate whether the problem is related to the analytical range. The low standard or the high standard having a response that is out of line with the others typically expresses this. In such situations, the analyst should determine whether the analytical range should be decreased by deleting the high or low standard. If the low standard is deleted, adjust the reporting limit. If the high standard is deleted, dilute samples appropriately. If the high standard shows saturation of the response, the calibration range must be adjusted. |

10.1.4.2. In all cases, the coefficient of determination (R^2) for all regression curves must be equal to or greater than 0.990.

10.2. Initial calibration verification

10.2.1. If the ICV fails to meet criteria [±10%], the analysis should be terminated, the problem corrected, the instrument recalibrated and the calibration reverified. (See Section 11.10 or 11.11 for required run sequence. Refer to the appropriate sections and Table VI for calibration verification process, acceptance criteria and corrective actions.

10.2.2. Not meeting this requirement may be indicative of serious system malfunction or inaccuracies in the standards used for the initial calibration curve or ICV standard. Corrective action must be taken (including reanalysis of the ICV, or analysis of a different ICV). Any decision to proceed with analysis of samples when the ICV is out-of-control must be taken with great care and in consultation with the QA department. Any such action must be documented in an NCM.

10.3. Initial Calibration Blank (ICB)

If the ICB fails to meet criteria [within +/- the RL from zero], the analysis should be terminated, the problem corrected, the instrument recalibrated and the calibration reverified. (Refer to the appropriate sections and Table VI for calibration verification process, acceptance criteria and corrective actions.)

10.4. Continuing Calibration Verification (CCV)

The CCV for both methods must fall within 10% of the true value for that solution with relative standard deviation <5% from replicate (minimum of two) exposures. Sample results may only be reported when bracketed by valid CCV/CCB pairs. If a mid-run CCV fails to meet criteria, the analysis for the affected element must be terminated, the problem corrected, the instrument recalibrated, the calibration verified and the affected samples reanalyzed. (Refer to the appropriate sections and Table VI for calibration verification process, acceptance criteria and corrective actions.)

10.5. Continuing Calibration Blank (CCB)

The CCB result must fall within +/- RL from zero. Sample results may only be reported when bracketed by valid CCV/CCB pairs. If a mid-run CCB fails, the analysis for the affected element must be terminated, the problem corrected, the instrument recalibrated, the calibration verified and the affected samples reanalyzed. (Refer to the appropriate sections and Table VI for calibration verification process, acceptance criteria and corrective actions.)

10.6. Contract Required Detection Limit Test of the ICP (CRI)

The CRI is a low-level CCV, sometimes referenced as the Low-Level Standard (LLS) of the analyte of interest. The CRA concentration is at or near the MDL. Since the EPA has not set acceptance limits for the CRA the CRA will be controlled to the client requirements or $\pm$ 25%. (Refer to Section 9.0 and Table II for calibration verification process, acceptance criteria and corrective actions.)

11. PROCEDURE

11.1. A minimum of two exposures for each standard, field sample and QC sample is required. The average of the exposures is reported. For Trace ICP analyses, the results of the sum channel must be used for reporting.

11.2. Prior to calibration and between each sample/standard the system is rinsed with the calibration blank solution. Triton-X can be added to the rinse solution to facilitate the rinse process.

11.3. The use of an autosampler for all runs is strongly recommended.

11.4. The use of automated QC checks through the instrument software is highly recommended for all calibration verification samples (ICV, CCV), blanks (ICB,CCB,PB), interference checks (ICSA,ICSAB) and field samples (linear range) to improve the data review process.

11.5. To facilitate the early identification of QC failures and samples requiring rerun it is strongly recommended that sample data be reviewed periodically throughout the run.

11.6. To facilitate the data review and reporting processes it is strongly recommended that all necessary dilutions and post digestion spikes be performed before closing out the instrument run.

11.7. For unattended overnight auto-runs it is strongly recommended that the frequency of ICSA/ICSAB analysis be increased to every 4 hours.

11.8. The use of an internal standard is recommended on the non-Trace ICPs.

11.9. Currently internal standards are not utilized at STL-St. Louis. For trace metals analysis STL- St. Louis uses both a high solids nebulizer and a mass flow controller.

The following procedural guidelines must be followed when using an internal standard:

11.9.1. Recommended internal standards are yttrium or scandium. (Note: Any element can be used that is not typically found in environmental samples at a high rate of occurrence.)

11.9.2. The internal standard (IS) must be added to every sample and standard at the same concentration. It is recommended that the IS be added to each analytical sample automatically through use of a third pump channel and mixing coil. Internal standards should be added to blanks, samples and standards in a like manner, so that dilution effects resulting from the addition may be disregarded.

11.9.3. The concentration of the internal standard should be sufficiently high to obtain good precision in the measurement of the IS analyte used for data correction and to minimize the possibility of correction errors if the IS analyte is naturally present in the sample.

11.9.4. The internal standard raw intensity counts must be printed on the raw data.

11.9.5. The analyst must monitor the response of the internal standard throughout the sample analysis run. This information is used to detect potential problems and identify possible background contributions from the sample (i.e., natural occurrence of IS analyte).

11.9.5.1. If the internal standard counts fall within ±30% of the counts observed in the ICB then the data is acceptable.

11.9.5.2. If the internal standard counts in the field samples are more than ±30% higher than the expected level, the field samples must then be screened without the addition of the internal standard.

11.9.5.3. If the internal standard element is not identified in the unspiked field sample at a level exceeding 10% of the level spiked, the data may be accepted.

11.9.5.4. If this IS analyte is detected in the unspiked field sample at a concentration greater than 10% of the spiked level then either:

- A different internal standard must be used.
- The IS concentration must be raised.
- The sample must be diluted and rerun.
- The analysis must be run without an internal standard (matrix matching must be substituted.)
- An alternate method of analysis (i.e., GFAA) must be applied.

11.10. The following analytical sequence must be used for this SOP:

Instrument Calibration
ICV
ICB
CRI
ICSA
ICSAB
CCV
CCB
10 samples
CCV
CCB
10 samples
CCV
CCB
Repeat sequence of up to 10 samples between CCV/CCB pairs as required to complete run
CCV
CCB
7 samples
CRI
ICSA
ICSAB
CCV
CCB

Refer to Quality Control Section 9.0 and Table VI (Appendix A) for quality control criteria.

11.11. The following run sequence provides an illustration of a mid-run CCV or CCB failure and the appropriate corrective action run sequence as described in Section 9.2.11:

Original Run:	Instrument Calibration	
	ICV	
	ICB	
	CRI	
	ICSA	
	ICSAB	
	CCV1	
	CCB1	
	10 samples	
	CCV2	
	CCB2	
	10 samples	
	CCV3	
	CCB3	
	10 samples	**
	CCV4 *	* Failure occurs at CCV4/CCB4
	CCB4 *	
	10 samples	** **Samples requiring rerun for affected analytes
	CCV5	
	CCB5	
	10 samples	**
	CCV6	
	CCB6	
	ICSA	
	ICSAB	
	CCV7	
	CCB7	
Reanalysis:	Recalibrate	
	CCV3	
	CCB3	
	10 samples	
	CCV4	
	CCB4	
	10 samples	
	CCV5	
	CCB5	
	10 samples	
	CCV6	
	CCB6	
	CRI	
	ICSA	
	ICSAB	
	CCV7	
	CCB7	

Notes: If the reanalysis is conducted under the same instrument setup conditions then it is not necessary to rerun the *CRI and* ICSA/ICSAB at the start of the reanalysis sequence as long as the 8 hour criteria are met. If reanalysis can't be initiated immediately or under the same run conditions then reanalysis must be conducted using the full analysis sequence as detailed in Section 11.10.

11.12. Full method required QC must be available for each wavelength used in determining reported analyte results.

11.13. All measurements must fall within the defined linear range where spectral interference correction factors are valid. Dilute and reanalyze all samples for required analytes that exceed the linear range or use an alternate wavelength for which QC data are established. If an interelement correction exists for an analyte which exceeds the linear range, the IEC may be inaccurately applied. Therefore, if an overrange interfering analyte causes a calculation error, as indicated by a "K" for the element of interest, in the sample the result may not be reported for that sample. For samples that initially give calculation errors a dilution will be performed to reduce interfering elements. Acid strength must be maintained in the dilution of samples.

In instances where TCLP analyses are requested by CLP protocols refer to the 6010B SOP (CORP-MT-0001) for **additional QC requirements.**

11.14. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

11.15. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Raw data reported in mg/L concentration off the curve.

12.2. ICV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(ICV)}{True(ICV)}\right)$$

12.3. CCV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(CCV)}{True(CCV)}\right)$$

12.4. Matrix Spike Recoveries are calculated according to the following equation:

$$\%R = 100\left(\frac{SSR - SR}{SA}\right)$$

Where:
SSR = Spike Sample Result
SR = Sample Result
SA = Spike Added

NOTE: When sample concentration is less than the instrument detection limit, use SR=0 for purposes of calculating % Recovery.

12.5. The relative percent difference (RPD) of sample duplicates are calculated according to the following equation:

$$RPD = 100\left[\frac{|DU1 - DU2|}{\left(\frac{DU1 + DU2}{2}\right)}\right]$$

Where:
DU1 = Sample result
DU2 = Sample duplicate result

12.6. The final concentration for a digested aqueous sample is calculated as follows:

$$mg/L = \frac{C \times V1 \times D}{V2}$$

Where:
C = Concentration (mg/L) from instrument readout
D = Instrument dilution factor
V1 = Final volume in liters after sample preparation
V2 = Initial volume of sample digested in liters

12.7. The final concentration determined in digested solid samples when reported on a dry weight basis is as follows:

$$mg/Kg, dry\ weight = \frac{C \times V \times D}{W \times S}$$

Where:
C = Concentration (mg/L) from instrument readout
D = Instrument dilution factor
V = Final volume in liters after sample preparation
W = Weight in Kg of wet sample digested
S = Percent solids/100

Note: A Percent Solids determination must be performed on a separate aliquot when dry weight concentrations are to be reported. If the results are to be reported on wet weight basis the "S" factor should be omitted from the above equation.

12.8. The LCS percent recovery is calculated according to the following equation:

$$\%R = 100\left(\frac{Found(LCS)}{True(LCS)}\right)$$

12.9. The serial dilution percent difference for each component is calculated as follows:

$$\%Difference = \frac{|I - S|}{I} \times 100$$

Where:
I = Sample result (Instrument reading)
S = Dilution test result (Instrument reading × 5)

12.10. Appropriate factors must be applied to sample values if dilutions are performed.

12.11. Sample results should be reported according to the following significant figure rules:

Significant Figures	Sample Results
2	<10
3	>10

13. DATA ASSESSMENT AND ACCEPTANCE CRITERIA

The following represent data assessment for samples and acceptance criteria for QC measures. Corrective actions for any failure in QC measures are shown in Section 0.

13.1. QC sample acceptance criteria

13.1.1. Method Blank.

13.1.1.1. No target analytes may be present in the method blank above the reporting limit with the exception of the common laboratory contaminates (Pb, Zn, Cu, and Fe).

13.1.2. Laboratory Control Sample (LCS).

13.1.2.1. Until in-house control limits are established, method 200.7 CLP-M will have a control limit of 80 – 120% on recovery applied.

13.1.2.2. The RPD for a LCS and LCSD will be compared to the MS/MSD RPD requirement of 20% (35% for soil).

13.1.2.3. All control analytes must be within established control limits for accuracy (%Recovery) and precision (RPD). Exceptions are allowed only with QA and project management approval.

13.1.2.4. It is desirable to have all non-control analytes within control limits, but it is expected that a small proportion will not be in control.

13.1.3. Matrix Spike/Matrix Spike Duplicate (MS/MSD).

13.1.3.1. Until in-house control limits are established, methods 6010B and 200.7 will have a control limit of 75 – 125% recovery will be applied for the MS/MSD.

13.1.3.2. The RPD for a MS/MSD pair is 20% (35% for soil).

13.1.3.3. All analytes should be within established control limits for accuracy (%Recovery) and precision (RPD). Deviations from this may be the results of matrix effects, which are confirmed by passing LCSs.

13.1.3.4. No specific corrective actions are required in the evaluation of the MS/MSD results provided that the batch LCS is in control. Analysts should use sound judgment in accepting MS/MSD results that are not within control limits, especially if the LCS results are borderline.

13.1.4. Low-Level Contract Required Detection Limit Test (CRI)

13.1.4.1. Since the EPA has not set acceptance limits for the CRI the CRI will be controlled to the client requirements or $\pm$ 25%.

13.1.5. Initial Calibration Verifications (ICV).

13.1.5.1. For analyses conducted under Method 200.7, the ICV result must fall within 10% of the true value for that solution with relative standard deviation <3% from replicate (minimum of two) exposures. If the ICV or fails to meet criteria, the analysis should be terminated, the problem corrected, the instrument recalibrated and the calibration reverified. (See Sections 11.10 and 11.11 for required run sequence).

13.1.6. Initial Calibration Blank (ICB).

13.1.6.1. An ICB is analyzed immediately following the ICV to monitor low level accuracy and system cleanliness. The ICB result must fall within +/- the RL from zero. If the ICB fails to meet criteria, the analysis should be terminated, the problem corrected, the instrument recalibrated and the calibration reverified. (See Section 11.11 for required run sequence).

13.1.7. Continuing Calibration Verifications (CCV)

13.1.7.1. The CCV for both methods must fall within 10% of the true value for that solution with relative standard deviation <5% from replicate (minimum of two) exposures. A CCB is analyzed immediately following each CCV. (See Section 11.11 for required run sequence.) The CCB result must fall within +/- RL from zero. Sample results may only be reported when bracketed by valid CCV/CCB pairs. If a mid-run CCV, the analysis for the affected element must be terminated, the problem corrected, the instrument recalibrated, the calibration verified and the affected samples reanalyzed.

13.1.8. Continuing Calibration Blank (CCB)

13.1.8.1. The CCB result must fall within +/- RL from zero. Sample results may only be reported when bracketed by valid CCV/CCB pairs. If a mid-run CCB fails, the analysis for the affected element must be terminated, the problem corrected, the instrument recalibrated, the calibration verified and the affected samples reanalyzed.

13.1.9. Interference Check Analysis (ICSA/ICSAB)

13.1.9.1. The ICSAB results for interferents must fall within 80 – 120% of the true value. ICSA results for the non-interfering elements with RLs < 10 µg/L must fall within ± 2x RL from zero. ICSA results for the non-interfering elements with RLs > 10 µg/L must fall within ± 1xRL from zero.

13.2. Sample result evaluation

13.2.1. Carryover. When a sample has a high response for a compound, there is a real possibility that some of the sample may carry over into the sample analyzed immediately afterward.

13.2.1.1. If a sample analyzed after a sample with high concentrations has negative results, carryover did not occur.

13.2.1.2. If a sample analyzed after a sample with high concentrations has positive results for the same analytes, or if the chromatographic profile resembles the previous sample, the results are questionable. This sample must be reanalyzed under conditions in which carryover can be confirmed to not have occurred.

13.2.2. Dilutions

13.2.2.1. If the response for any compound exceeds the working range of the analytical system, a dilution of the extract is prepared and analyzed. An appropriate dilution should be in the upper half of the calibration range.

13.2.2.2. If the initial diluted run has no hits or hits below 20% of the calibration range and the matrix allows for analysis at a lesser dilution, then the sample must be reanalyzed at a dilution targeted to bring the largest hit in the upper half of the calibration range.

14. CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

14.1. Method Blank.

14.1.1. The samples in the batch associated to the defective method blank are evaluated:

14.1.1.1. If the analyte found in the method blank is confirmed to not be present in any of the associated samples at any level, the contamination did not affect those samples. This represents a non-impact situation and no specific corrective action is taken. A nonconformance memo is written to notify project management of the situation for evaluation against project requirements.

14.1.1.2. If the analyte is a common laboratory contaminant (copper, iron, lead (Trace only) or zinc) the data may be reported with qualifiers if the concentration of the analyte in the method blank is less than two times the RL. **Such action must be taken in consultation with the client and must be addressed with a Non Conformance Memorandum, (NCM) in the project narrative.**

14.1.1.3. If the analyte found in the method blank is confirmed to be present in one or more of the associated samples, the concentrations are compared.

14.1.1.3.1. Repreparation and reanalysis of all samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples (see exception noted in 14.1.1.2).

14.1.1.3.2. If there is no analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. **Such action must be addressed in an NCM.**

14.1.1.4. If the criteria of 14.1.1.3 are not met and reanalysis is not possible, then the sample data must be qualified. **This anomaly must be addressed with a NCM in the project narrative and the client must be notified.**

14.1.1.5. If the analyte found in the method blank is not one of the common laboratory contaminants (Fe, Pb [Trace only], Cu, and Zn) and routine corrective actions do not return the system to service, the analytical system must be placed out of service until the source of the problem is identified and corrected.

14.2. Laboratory Control Sample

14.2.1. If any control analyte is out of control for accuracy (% Recovery), the associated samples are evaluated.

14.2.1.1. If the recovery is biased high and the associated samples have results <RL for that analyte, a non-impact situation ensues. A nonconformance memo is written to notify project management of the situation for evaluation against project requirements. **Based upon client consultation, the samples may have to be rerun.**

14.2.1.2. If the recovery is biased low and the associated samples have positive results for that analyte, a minimal impact situation ensues. A nonconformance memo is written to notify project management of the situation for evaluation against project requirements. **Based upon client consultation, the samples (and batch) may have to be rerun.**

14.2.1.3. If the recovery is biased high and the associated samples have positive results for that analyte, the prescribed corrective action is redigestion of the samples and reanalysis of the batch.

14.2.1.4. If the recovery is biased low and the samples have no positive results for that analyte, the prescribed corrective action is redigestion of the samples and reanalysis of the batch.

14.2.2. If any control analyte is out of control for precision (RPD), the associated samples are evaluated. All decisions about corrective action(s) are made in consultation with the project manager.

14.2.2.1. If there are no positive results in one or more samples, a non-impact situation ensues for those samples. A nonconformance memo is written to notify project management of the situation for evaluation against project requirements.

14.2.2.2. If there are positive results for one or more analytes, the likelihood of poor reproducibility increases and corrective action must be evaluated. A nonconformance memo is written to notify project management of the situation for a project decision on whether the affected sample(s) should be reanalyzed.

14.2.3. In the event that an MS/MSD analysis is not possible, a Laboratory Control Sample Duplicate (LCSD) must be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits

14.3. Matrix Spike/Matrix Spike Duplicate

14.3.1. If any control analyte is out of control for accuracy (% Recovery), the associated samples are evaluated.

14.3.1.1. If any analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. For method 200.7 CLP-M, control limits of 75 - 125% recovery and 20% (35% for soil) RPD or historical acceptance criteria must be applied to the MS/MSD. If the LCS recovery is within limits, then the laboratory operation is in control and the results may be accepted. If the recovery of the LCS is outside limits corrective action must be taken. Corrective action will include repreparation and reanalysis of the batch. MS/MSD results which fall outside the control limits must be addressed in the narrative.

14.3.1.2. If the native analyte concentration in the MS/MSD exceeds 4x the spike level for that analyte, the recovery data are reported as NC (i.e., not calculated). If the reporting software does not have the ability to report NC then the actual recovery must be reported and addressed in the case narrative."

14.3.1.3. If an MS/MSD is not possible due to limited sample volume then a laboratory control sample duplicate (LCSD) should be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.

14.4. Low-Level Contract Required Detection Limit Test (CRI)

14.4.1. If the CRI fails the system will be evaluated and checked for errors. A failure of the CRI does not necessitate reanalysis.

14.5. Initial Calibration Verification (ICV)

14.5.1. If the ICV or fails to meet criteria, the analysis should be terminated, the problem corrected, the instrument recalibrated and the calibration reverified. (See Section 11.11 for required run sequence).

14.6. Initial Calibration Blank (ICB)

14.6.1. If the ICB or fails to meet criteria [within +/- the RL from zero], the analysis should be terminated, the problem corrected, the instrument recalibrated and the calibration reverified.

14.7. Continuing Calibration Verification (CCV)

14.7.1. The CCV must fall within 10% of the true value for that solution with relative standard deviation <5% from replicate (minimum of two) exposures. Sample results may only be reported when bracketed by valid CCV/CCB pairs. If a mid-run CCV, the analysis for the affected element must be terminated, the problem corrected, the instrument recalibrated, the calibration verified and the affected samples reanalyzed.

14.8. Continuing Calibration Blank (CCB)

14.8.1. The CCB result must fall within +/- RL from zero. Sample results may only be reported when bracketed by valid CCV/CCB pairs. If a mid-run CCB fails, the analysis for the affected element must be terminated, the problem corrected, the instrument recalibrated, the calibration verified and the affected samples reanalyzed.

14.9. Interference Check Analysis (ICSA/ICSAB)

14.9.1. If any results for the interferents for the ICSA/ICSAB are out of control, the associated samples are evaluated.

14.9.1.1. If any ICSAB interferent results fail criteria, the analysis should be terminated, the problem corrected, the instrument recalibrated and the samples rerun.

14.9.1.2. If the ICSA results for the non-interfering elements do not fall within +/- 2x RL (RL ≤10) or ± 1xRL (RL>10) from zero the field sample data must be evaluated as follows:

14.9.1.2.1. If the non-interfering element concentration in the ICSA is the result of contamination versus a spectral interference, and this reason is documented, the field sample data can be accepted.

14.9.1.2.2. If the affected element was not required then the sample data can be accepted.

14.9.1.2.3. If the interfering elements are not present in the field sample at a concentration which would result in a false positive or negative result greater than +/- 2x RL from zero then the field sample data can be accepted.

14.9.1.2.4. If the interfering element is present in the field sample at a level which would result in a false analyte signal greater than ± 2x RL from zero, the data can be accepted only if the concentration of the affected analyte in the field sample is more than 10x the analyte signal in the ICSA.

14.9.1.2.5. If the data does not meet the above conditions then the IECs must be re-evaluated and corrected if necessary and the affected samples reanalyzed or the sample results manually corrected through application of the new IEC to the raw results. If the results are recalculated manually the calculations must be clearly documented on the raw data.

15. CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

15.1. Method blanks

15.1.1. If there is insufficient sample to perform re-analysis, the analyst must notify the project manager for consultation with the client. In this situation, all associated samples are flagged with a "B" qualifier (or appropriate qualifier) and appropriate comments in the narrative.

15.2. LCS

15.2.1. If the batch is not re-extracted and reanalyzed, the reasons for accepting the batch must be clearly presented in the project records and the report. Acceptable matrix spike recoveries are not sufficient justification to preclude re-extraction of the batch.

15.2.2. If re-extraction and reanalysis of the batch is not possible due to limited sample volume or other constraints, the LCS is reported, all associated samples are flagged, and appropriate comments are made in a narrative to provide further documentation.

15.3. Insufficient sample

15.3.1. If there is insufficient sample to repeat the analysis, the project manager is notified via NCM for consultation with the client.

16. METHOD PERFORMANCE

16.1. Each laboratory must have initial demonstration of performance data on file for each analyte of interest as described in Section 9.0.

16.2. Refer to Tables I, IA & II in Appendix A for the list of Method 200.7 CLP-M analytes as well as additional analytes that may be analyzed using this SOP.

16.3. Method performance is determined by the analysis of matrix spike and matrix duplicate samples as well as preparation blanks and laboratory control samples. The matrix spike recovery should fall within +/- 25% and the matrix duplicates should compare within 20% RPD. Preparation blanks must meet the criteria specified in Section 9.2.5. The laboratory control samples should recover within 20% of the true value for aqueous LCS and within the control limits supplied by the manufacturer of the soil LCS.

16.4. Training Qualification:

The group/team leader or the supervisor has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

17. POLLUTION PREVENTION

This procedure will be carried out in a manner consistent with all applicable federal state and local regulations regarding pollution control and prevention. Specific controls due to the accidental release of hazardous materials can be found in the Chemical Hygiene Plan and facility attachments.

18. WASTE MANAGEMENT

18.1. Waste generated in this procedure will be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Coordinator should be contacted if additional information is required.

18.2. Standards should be purchased and prepared in volumes consistent with laboratory use to minimize the volume of expired standards to be disposed.

19. REFERENCES

19.1. USEPA Statement of Work for Inorganics Analysis, ILMO3.0.

19.2. CORP-MT-0001, Inductively Coupled Plasma-Atomic Emission Spectroscopy, Spectrometric Method of Trace Element Analysis, Method 6010B and Method 200.7.

20. CLARIFICATIONS, MODIFICATIONS AND ADDITIONS TO REFERENCE METHOD

20.1. Modifications/Interpretations from reference method

20.1.1. The method calls for the use of ASTM Type II water. This SOP specifies the use of a Millipore DI system or equivalent to produce reagent water. This SOP requires that reagent water must be free of the analytes of interest as demonstrated through the analysis of preparation blanks.

20.2. Modifications from previous SOP

20.2.1. Refer to version2.1 of this SOP.

20.3. Facility Specific SOPs.

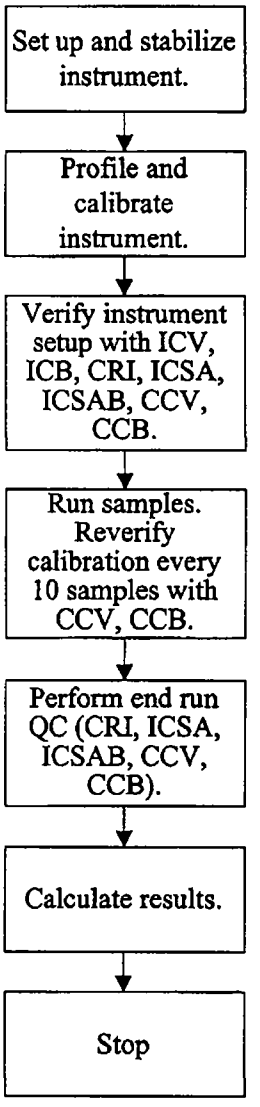
20.3.1. This SOP has been modified to depict the standard operating procedures at STL-St. Louis.

20.4. Documentation and Record Management

The following documentation comprises a complete ICP raw data package:

- Raw data (direct instrument printout signed by analyst).
- Relevant sample preparation benchsheets.
- Run log printout from instrument software where this option is available (TJA) or manually generated run log (i.e., Ward WSL printout).
- Data review checklist - See Appendix B.
- Standards documentation (including prep date, source, and lot #).
- Nonconformance summary (if applicable).

20.5. Flow Diagram



APPENDIX A

TABLES

TABLE I. Method 200.7 CLP-M Analyte List

ELEMENT	Symbol	CAS #	CRDL (ug/L) Water	CRDL (mg/kg) Soil
Aluminum	Al	7429-90-5	200	40
Antimony	Sb	7440-36-0	60	12
Arsenic	As	7440-38-2	10*	2*
Barium	Ba	7440-39-3	200	40
Beryllium	Be	7440-41-7	5	1
Cadmium	Cd	7440-43-9	5	1
Calcium	Ca	7440-70-2	5000	1000
Chromium	Cr	7440-47-3	10	2
Cobalt	Co	7440-48-4	50	10
Copper	Cu	7440-50-8	25	5
Iron	Fe	7439-89-6	100	20
Lead	Pb	7439-92-1	3*	0.6*
Magnesium	Mg	7439-95-4	5000	1000
Manganese	Mn	7439-96-5	15	3
Nickel	Ni	7440-02-0	40	8
Potassium	K	7440-09-7	5000	1000
Selenium	Se	7782-49-2	5*	1*
Silver	Ag	7440-22-4	10	2
Sodium	Na	7440-23-5	5000	1000
Thallium	Tl	7440-28-0	10*	2*
Vanadium	V	7440-62-2	50	10
Zinc	Zn	7440-66-6	20	4

* Analyte is reported at five times IDL.

TABLE IA. Method 200.7 CLP-M Trace ICP Analyte List

ELEMENT	Symbol	CAS #	CRDL (ug/L) Water	CRDL (mg/kg) Soil
Arsenic	As	7440-38-2	10	2
Lead	Pb	7439-92-1	3	0.6
Selenium	Se	7782-49-2	5	1
Thallium	Tl	7440-28-0	10	2

TABLE II. Non-Routine Analyte List

ELEMENT	Symbol	CAS #	Reporting Limit (ug/L) Water	Reporting Limit (mg/kg) Soil
Boron	B	7440-42-8	200	40
Lithium	Li	7439-93-1	50	10
Molybdenum	Mo	7439-98-7	40	8
Phosphorous	P	7723-14-0	300	60
Silicon	Si	7631-86-9	500	100
Strontium	Sr	7740-24-6	50	10
Tin	Sn	7440-31-5	100	20
Titanium	Ti	7440-03-26	50	10
Bismuth	Bi	7440-06-99	200	40
Zirconium	Zr	7440-06-77	100	20
Tungsten	W	7440-03-37	500	100
Tellurium	Te	1349-48-09	500	100
Thorium	Th	7440-02-91	500	100
Uranium	U	7440-06-11	500	100
Palladium	Pd	7440-00-53	100	20

NOTE: Analysis of all elements listed may not be available at all STL facilities.

TABLE III. Matrix Spike and Aqueous Laboratory Control Sample* Levels

ELEMENT	LCS Level (ug/L)	Matrix Spike Level (ug/L)
Aluminum	1000	2000
Antimony	1000	500
Arsenic	1000	2000
Barium	1000	2000
Beryllium	1000	50
Cadmium	1000	50
Calcium	20000	50000
Chromium	1000	200
Cobalt	1000	500
Copper	1000	250
Iron	1000	1000
Lead	1000	500
Lithium	2000	2000
Magnesium	20000	50000
Manganese	1000	500
Molybdenum	1000	1000
Nickel	1000	500
Phosphorous	10000	10000
Potassium	20000	50000
Selenium	1000	2000
Silver	250	50
Sodium	20000	50000
Strontium	1000	1000
Thallium	1000	2000
Vanadium	1000	500
Zinc	1000	500
Boron	2000	2000
Silicon	1000	2000
Tin	2000	2000
Titanium	2000	2000
Bismuth	2000	2000
Zirconium	2000	2000
Tellurium	2000	2000
Thorium	2000	2000
Uranium	2000	2000

* Analyses conducted under direct contract to the USEPA CLP must be performed using the LCS materials supplied by the USEPA

TABLE IV. ICP Calibration and Calibration Verification Standards

Element	Calibration Level	CRDL (ug/L)	CRI (ug/L)	ICV (ug/L)*	CCV (ug/L)
Aluminum	100000	200	400	50000	40000
Antimony	10000	60	120	5000	4000
Arsenic	10000	10	20**	5000	4000
Barium	10000	200	400	5000	4000
Beryllium	10000	5	10	5000	4000
Cadmium	10000	5	10	5000	4000
Calcium	50000	5000	10000	25000	20000
Chromium	10000	10	20	5000	4000
Cobalt	10000	50	100	5000	4000
Copper	10000	25	50	5000	4000
Iron	100000	100	200	50000	40000
Lead	10000	3	6**	5000	4000
Lithium	10000	50	100	5000	4000
Magnesium	50000	5000	10000	25000	20000
Manganese	10000	15	30	5000	4000
Molybdenum	10000	40	80	5000	4000
Nickel	10000	40	80	5000	4000
Phosphorous	10000	300	600	5000	4000
Potassium	100000	5000	10000	50000	40000
Selenium	10000	5	10**	5000	4000
Silver	2000	10	20	1250	1000
Sodium	100000	5000	10000	50000	40000
Strontium	10000	50	100	5000	4000
Thallium	10000	10	20**	5000	4000
Vanadium	10000	50	100	5000	4000
Zinc	10000	20	40	5000	4000
Boron	10000	200	400	5000	4000
Silicon	10000	500	1000	5000	4000
Tin	10000	100	200	5000	4000
Titanium	10000	50	100	5000	4000
Bismuth	10000	200	400	5000	4000
Zirconium	10000	100	200	5000	4000
Tellurium	10000	500	1000	5000	4000
Thorium	10000	500	1000	5000	4000
Uranium	10000	500	1000	5000	4000

*Analyses conducted under direct contract to the USEPA CLP must be performed using the ICV materials supplied by the USEPA.

**Analytes should be spiked at two times the IDL or two times the CRDL, whichever is greater when non ICP trace analysis are performed.

- **TABLE V. Interference Check Sample Concentrations***

Element	ICSA (ug/L)	ICSAB (ug/L)**
Aluminum	500000	500000
Antimony	-	1000
Arsenic	-	1000
Barium	-	500
Beryllium	-	500
Cadmium	-	1000
Calcium	500000	500000
Chromium	-	500
Cobalt	-	500
Copper	-	500
Iron	200000	200000
Lead	-	1000
Magnesium	500000	500000
Manganese	-	500
Molybdenum	-	1000
Nickel	-	1000
Potassium	-	20000
Selenium	-	1000
Silver	-	1000
Sodium	-	20000
Thallium	-	1000
Vanadium	-	500
Zinc	-	1000
Tin	-	1000

* Analyses conducted under direct contract to the USEPA CLP must be performed using the ICS solutions supplied by the USEPA.

**Custom solutions contain analytes common to all STL facilities. Non-routine elements not listed above *may be added into the ICSAB as a client specific requirement.*

TABLE VI. Summary Of Quality Control Requirements

QC PARAMETER	FREQUENCY *	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
CRI	Beginning and end of every analytical run.	None	None
ICV	Beginning of every analytical run.	90-110% recovery.	Terminate analysis; Correct the problem; Recalibrate.
ICB	Immediately after each ICV.	The result must be within +/- CRDL from zero.	Terminate analysis; Correct the problem; Recalibrate.
CCV	Beginning and end of run and every 10 samples or every 2 hours, whichever is more frequent.	90-110% recovery.	Terminate analysis; Correct the problem; Recalibrate the instrument, reverify calibration and rerun all samples since the last acceptable CCV.
CCB	Immediately following each CCV.	The result must be within +/- CRDL from zero.	Terminate analysis; Correct the problem; Recalibrate the instrument, reverify calibration and rerun all samples since the last acceptable CCB.
ICSA	Beginning and end of every run and every 8 hours.	Analyte results must be within +/- 2x CRDL from zero.	See Section 9.2.12.
ICSAB	Immediately following each ICSA.	Results must be within 80 - 120% recovery.	See Section 9.2.12.

* See Sections 11.10 for exact run sequence to be followed.

TABLE VI. Summary of Quality Control Requirements (Continued)

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Serial Dilution	One per SDG or group of samples of a similar matrix type whichever is more frequent.	For samples > 50x IDL, dilutions must agree within 10%.	Narrate the possibility of physical or chemical interference.
Preparation Blank	One per sample preparation batch of up to 20 samples.	The result must be within ± CRDL from zero. Sample results greater than 10x the blank concentration or samples for which the contaminant is < CRDL do not require redigestion or reanalysis.	Redigest and reanalyze samples. Note exceptions under criteria section. See Section 9.2.5 for additional requirements.
Laboratory Control Sample (LCS)	One per sample preparation batch of up to 20 samples.	Aqueous LCS must be within 80 - 120% recovery. (exception: Ag and Sb). Soil LCS must be within the limits provided by the supplier.	Terminate analysis; Correct the problem; Redigest and reanalyze all samples associated with the LCS.
Matrix Spike	One per SDG or each group of up to 20 samples of a similar matrix type.	75 - 125 % recovery (exception: Ag).	Flag the data; no flag required if the sample level is > 4x the spike added. (See post digest spike).
Matrix Duplicate	See Matrix Spike	RPD ± 20% (See MS).	Flag the data.
Post Digest Spike (PDS)	When MS recovery falls outside 75 -125% (Exception: Ag).	None	None

APPENDIX B

ICP DATA REVIEW CHECKLIST

Appendix B: ICP Data Review Checklist

Run/Project Information:

Run Date: _____ **Analyst:** _____ **Instrument:** _____
Prep Batches Run: _____

Circle Methods used: 6010B / 200.7: CORP-MT-0001STL Rev 2.2
CLP ILMO3.0/4.0 : CORP-MT-0002STL Rev 2.2

Review Items

A. Calibration/Instrument Run QC	Yes	No	N/A	2nd Level
1. Instrument calibrated per manufacturer's instructions and at SOP specified levels ?				
2. ICV/CCV analyzed at appropriate frequency and within control limits ? (6010B, CLP = 90 - 110%, 200.7 = 95 -105%[ICV])				
3. ICB/CCB analyzed at appropriate frequency and within +/- RL or +/- CRDL (CLP) ?				
4. CRI analyzed? (for CLP only)				
5. ICSA/ICSAB run at required frequency and within SOP limits ?				
B. Sample Results				
1. Were samples with concentrations > the linear range for any parameter diluted and reanalyzed ?				
2. All reported results bracketed by in control QC ?				
3. Sample analyses done within holding time ?				
C. Preparation/Matrix QC				
1. LCS done per prep batch and within QC limits ?				
2. Method blank done per prep batch and < RL or CRDL (CLP) ?				
3. MS run at required frequency and within limits ?				
4. MSD or DU run at required frequency and RPD within SOP limits ?				
5. Dilution Test done per prep batch (or per SDG for CLP) ?				
6. Post digest spike analyzed if required (CLP only) ?				
D. Other				
1. Are all nonconformances documented appropriately ?				
2. Current IDL/LR/IEC data on file ?				
3. Calculations checked for error ?				
4. Transcriptions checked for error ?				
5. All client/project specific requirements met ?				
6. Date/time of analysis verified as correct ?				

Analyst: _____ **Date:** _____
Comments: _____

2nd Level Reviewer : _____ **Date:** _____
Comments: _____

APPENDIX C

TROUBLESHOOTING GUIDE

APPENDIX C. TROUBLESHOOTING GUIDE

Problem	Possible Cause/ Solution
High Blanks	Increase rinse time Clean or replace tip Clean or replace torch Clean or replace sample tubing Clean or replace nebulizer Clean or replace mixing chamber Lower Torch
Instrument Drift	RF not cooling properly Vacuum level is too low Replace torch (Crack) Clean or replace nebulizer (blockage) Check room temperature (changing) Replace pump tubing Room humidity too high Clean torch tip (salt buildup) Check for argon leaks Adjust sample carrier gas Replace PA tube
Erratic Readings, Flickering Torch or High RSD	Check for argon leaks Adjust sample carrier gas Replace tubing (clogged) Check drainage(back pressure changing) Increase uptake time (too short) Increase flush time (too short) Clean nebulizer, torch or spray chamber Increase sample volume introduced Check that autosampler tubes are full Sample or dilution of sample not mixed Increase integration time (too short) Realign torch Reduce amount of tubing connectors
Intensity Ratio Outside Limits or Low Sensitivity	Plasma conditions changed Clean nebulizer, torch or spray chamber Replace tubing (clogged) Realign torch Check IECs
Standards reading twice normal absorbance or concentration	Incorrect standard used Incorrect dilution performed

APPENDIX D

CONTAMINATION CONTROL GUIDELINES

APPENDIX D. CONTAMINATION CONTROL GUIDELINES

The following procedures are strongly recommended to prevent contamination:

All work areas used to prepare standards and spikes should be cleaned before and after each use.

All glassware should be washed with detergent and tap water and rinsed with 1:1 HCl followed by a 1:1 nitric acid followed by deionized water.

Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.

Powdered or Latex Gloves must not be used in the metals laboratory since the powder contains silica and zinc as well as other metallic analytes. Only vinyl or nitrile gloves should be used in the metals laboratory.

Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.

The following are helpful hints in the identification of the source of contaminants:

Yellow pipet tips and volumetric caps can sometimes contain cadmium.

Some sample cups have been found to contain lead.

The markings on glass beakers have been found to contain lead. If acid baths are in use for glassware cleaning, they should be periodically checked for contaminants since contaminant concentrations will increase over time.

New glassware especially beakers can be a source of silica and boron.

Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.

Improper cleaning of glassware can cause contamination.

Latex gloves contain over 500 ppb of zinc.

APPENDIX E

PREVENTIVE MAINTENANCE

APPENDIX E. PREVENTIVE MAINTENANCE

A maintenance log is used to record when maintenance is performed on instruments. When an instrument problem occurs indicate the date, time and instrument number, then identify the problem and corrective action in the maintenance log.

The following procedures are required to ensure that that the instrument is fully operational.

Daily	Change sample pump tubing Check argon gas supply level Check rinse solution and fill if needed Check waste containers and empty if needed Check sample capillary tubing is clean and in good condition Check droplet size to verify nebulizer is not clogged. Check sample flow for cross flow nebulizer Check intensity ratio Check pressure for vacuum systems
As Needed	Clean plasma torch assembly to remove accumulated deposits Clean nebulizer and drain chamber; keep free-flowing to maintain optimum performance Replace peristaltic pump tubing, sample capillary tubing and autosampler sipper probe Apply silicon spray on autosampler tracks. Check oil for vacuum systems.
Monthly	Check water level in coolflow. Clean air filters on back of power unit to remove dust
Bi-Yearly	Replace coolant water filter (may require more or less frequently depending on quality of cooling water).

Attachment QAPP-C2

Inductively Coupled Plasma-Atomic Emission Spectroscopy, Spectrographic Method for Trace Element Analysis Method 6010B and EPA Method 200.

SOP No.:	CORP-MT-0001STL
Revision No.:	2.2
Revision Date:	10/31/00
Page:	1 of 46
Implementation Date:	04/24/01

Controlled Copy Number: _____

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STL ST. LOUIS STANDARD OPERATING PROCEDURE

TITLE: INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION SPECTROSCOPY, SPECTROMETRIC METHOD FOR TRACE ELEMENT ANALYSES, SW-846 METHOD 6010B AND EPA METHOD 200.7

(SUPERSEDES: CORP-MT-0001STL, REVISION 2.1)

Prepared by: _____

Approved by: _____
Technical Specialist

Approved by: _____
Quality Assurance Manager

Approved by: _____
Environmental Health and Safety Coordinator

Approved by: _____
Laboratory Director

1. SCOPE AND APPLICATION

1.1. This procedure describes the analysis of trace elements including metals in solution by Inductively Coupled Plasma -Atomic Emission Spectroscopy (ICP-AES) using SW-846 Method 6010B and EPA Method 200.7 . Table I of Appendix A lists the elements appropriate for analysis by Methods 6010B and 200.7. Additional elements may be analyzed under Methods 6010B and 200.7 provided that the method performance criteria presented in Section 16.0 are met.

1.2. Method 6010B is applicable to the determination of dissolved, suspended, total recoverable and total elements in ground water, aqueous samples, soils, sludges, wastes, sediments, and TCLP, EP and other leachates/extracts. All matrices require digestion prior to analysis with the exception of analyses for dissolved metals in filtered and acidified aqueous samples. Although digestion is not specifically required by the method, some clients and regulators may require digestion of **dissolved samples** and this must be clarified and documented before project initiation. Silver concentrations must be below 2.0 mg/L in aqueous samples and 100 mg/kg in solid matrix samples. Precipitation may occur in samples where silver concentrations exceed these levels and lead to the generation of erroneous data.

1.3. Method 200.7 is applicable to the determination of dissolved, suspended, total recoverable, and total elements in water, waste water, and solid wastes. All matrices require digestion prior to analysis with the exception of analyses for dissolved metals in filtered and acidified aqueous samples if the criteria in Section 11.1 are met. Silver concentrations must be below 0.1 mg/L in aqueous samples and 50 mg/kg in solid matrix samples.

1.4. Method 6010B is applicable to the determination of dissolved, suspended, total recoverable and total elements in ground water, aqueous samples, soils, sludges, wastes, sediments, and TCLP, EP and other leachates/extracts. All matrices require digestion prior to analysis with the exception of analyses for dissolved metals in filtered and acidified aqueous samples. Although digestion is not specifically required by the method, some clients and regulators may require digestion of **dissolved samples** and this must be clarified and documented before project initiation. Silver concentrations must be below 2.0 mg/L in aqueous samples and 100 mg/kg in solid matrix samples. Precipitation may occur in samples where silver concentrations exceed these levels and lead to the generation of erroneous data.

1.5. This SOP is based on SW-846 Method 6010B and EPA Method 200.7

1.6. The Target Analyte List and their reporting limits are listed in Table 1 of Appendix A. Reporting limits will be proportionately higher for sample extracts that require dilution and for soil samples that require concentration adjustments to account for % moisture.

1.7. ICP analysis provides for the determination of metal concentrations over several orders of magnitude. Detection limits, sensitivity and optimum concentration ranges of the metals will vary with the matrices and instrumentation used. For instance, in comparison to conventional ICP technique, ICP-Trace can achieve detection levels comparable to those determined using the graphite furnace atomic absorption spectroscopy (GFAAS) technique.

1.8. State-specific requirements may take precedence over this SOP for drinking water sample analyses

1.9. Method detection limits are maintained in the Information Management System (QuantIMS). Because of their dynamic nature, they are not specifically listed in this document, but can be retrieved at any time using TraQAr tools.

1.10. Quality control limits (accuracy and precision for spikes) are also maintained in QuantIMS, and are also dynamic. Therefore, they are not specifically listed in this document, but can be retrieved at any time using TraQAr tools.

1.11. Additional compounds may be amenable to this method. The minimum requirement for non-standard analytes is that the reporting limit be set at the lowest required concentration that can actually be detected by the instrument, and when an MDL study can not be conducted, the MDL be set equal to the reporting limit.

2. SUMMARY OF METHOD

2.1. This method describes a technique for the determination of multi elements in solution using sequential or simultaneous optical systems and axial or radial viewing of the plasma. The basis of the method is the measurement of atomic emission by an optical spectroscopic technique. Samples are nebulized and the aerosol that is produced is transported to the plasma torch where excitation occurs. Characteristic atomic-line emission

spectra are produced by a radio frequency inductively coupled plasma (ICP). The spectra are dispersed by a grating spectrometer and the intensities of the emission lines are monitored by photomultiplier tubes. The photocurrents from the photomultiplier tubes are processed and controlled by a computer system. A background correction technique is required to compensate for variable background contribution to the determination of trace elements. Background must be measured adjacent to analyte lines during analysis. The position selected for the background intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. The position used must be free of spectral interferences and reflect the same change in background intensity as occurs at the analyte wavelength measured. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result. The possibility of additional interferences should also be recognized and appropriate actions taken.

2.2. Refer to the appropriate SOPs for details on sample preparation methods.

3. DEFINITIONS

3.1. See policy QA-003 for definitions

3.2. Dissolved Metals: Those elements which will pass through a 0.45 um membrane filter. (Sample is acidified after filtration)

3.3. Suspended Metals: Those elements which are retained by a 0.45 um filter.

3.4. Total Metals: The concentration determined on an unfiltered sample following vigorous digestion.

3.5. Total Recoverable Metals: The concentration determined on an unfiltered sample following treatment with hot, dulute mineral acid.

4. INTERFERENCES

4.1. Spectral, physical and chemical interference effects may contribute to inaccuracies in the determinations of trace elements by ICP. Spectral interferences are caused by:

- Overlap of a spectral line from another element.
- Unresolved overlap of molecular band spectra.
- Background contribution from continuous or recombination phenomena.
- Stray light from the line emission of high concentration elements.

4.1.1. A background correction technique is required to compensate for variable background contribution to the determination of trace elements. Background correction is not required in cases where a background corrective measurement would actually degrade the analytical result.

4.1.2. Inter-element correction factors (IECs) are necessary to compensate for spectral overlap. Inter-element interferences occur when elements in the sample emit radiation at wavelengths so close to that of the analyte that they contribute significant intensity to the analyte channel. If such conditions exist, the intensity contributed by the matrix elements will cause an excessively high (or sometimes low) concentration to be reported for the analyte. Inter-element corrections IECs must be applied to the analyte to remove the effects of these unwanted emissions.

4.1.3. Physical interferences are generally considered to be effects associated with sample transport, nebulization and conversion within the plasma. These interferences may result in differences between instrument responses for the sample and the calibration standards. Physical interferences may occur in the transfer of solution to the nebulizer (e.g., viscosity effects), at the point of aerosol formation and transport to the plasma (e.g., surface tension) or during excitation and ionization processes within the plasma itself. Changes in viscosity and surface

tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If physical interferences are present, dilution of the sample, use of a peristaltic pump, mass flow controller, use of an internal standard and/or use of a high solids nebulizer can reduce the effect.

4.1.4. Chemical interferences are characterized by molecular compound formation, ionization effects and solute vaporization effects. Normally these effects are not significant with the ICP technique but if observed can be minimized by buffering the sample, matrix matching or standard addition procedures.

5. SAFETY

5.1. Procedures shall be carried out in a manner that protects the health and safety of all associates.

5.2. The Chemical Hygiene Plan (CHP) gives details about the specific health and safety practices which are to be followed in the laboratory area. Personnel must receive training in the CHP, including the written Hazard Communication plan, prior to working in the laboratory. Consult the CHP, the Health and Safety Policies and Procedures Manual, and available Material Safety Data Sheets (MSDS) prior to using the chemicals in the method.

5.3. Consult the Health and Safety Policies and Procedures Manual for information on Personal Protective Equipment. Eye protection that satisfies ANSI Z87.1 (as per the Chemical Hygiene Plan), laboratory coat and appropriate gloves must be worn while samples, standards, solvents and reagents are being handled. Disposable gloves that have become contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.4. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the Material Safety Data Sheets (MSDS) files maintained in the laboratory.

5.4.1. The following materials are known to be **corrosive:**

Sulfuric acid, hydrochloric acid, nitric acid and hydrofluoric acid. (NOTE: sulfuric and hydrofluoric acids are used in cleaning the ICP torch and hydrofluoric acid is also commonly used in air toxics preparations.)

5.4.2. The following materials are known to be **oxidizing agents:**

Nitric acid and hydrogen peroxide

5.4.3. The plasma emits strong UV light and is harmful to vision. **NOTE: Avoid looking directly at the plasma.**

5.4.4. The RF generator produces strong radio frequency waves, most of which are unshielded. People with pacemakers should not go near the instrument while in operation.

5.5. Exposure to chemicals will be maintained **as low as reasonably achievable,** therefore, unless they are known to be non-hazardous, all samples will be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Metal digestates can be processed outside of a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.

5.6. The preparation of reagents will be conducted in a fume hood with the sash closed as far as the operations will permit. When possible the preparation of standards should be conducted in a fume hood, in other case, the standards will be made in an area free of potential contaminating agents.

5.7. All work must be stopped in the event of a known, or potential compromise to the health or safety of any associate. The situation must be reported **immediately** to a laboratory supervisor.

5.8. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices outlined in the Health and Safety Manual. These employees must have training on the hazardous waste disposal practices initially upon assignment of these tasks, followed by an annual refresher training.

5.9. The use of hydrofluoric acid requires special safety precautions. Consult the facility EH&S Professional and laboratory supervisor for guidance.

6. EQUIPMENT AND SUPPLIES

6.1. Inductively Coupled Plasma Atomic Emission Spectrometer equipped with autosampler and background correction.

6.2. Radio Frequency Generator

6.3. Argon gas supply, welding grade or better

6.4. Cool-flow or appropriate water cooling device

6.5. Peristaltic Pump

6.6. Calibrated automatic pipettes or Class A glass volumetric pipettes

6.7. Class A volumetric flasks

6.8. Autosampler tubes

7. REAGENTS AND STANDARDS

7.1. Reagents

7.1.1. All reagent preparation is documented in the reagent logbook. All reagents are labeled with their unique ID, name (and concentration, if applicable) of the reagent, the date prepared and the expiration date.

7.1.2. Concentrated nitric acid (HNO3), trace metal grade or better

7.1.3. Concentrated hydrochloric acid (HCl), trace metal grade or better

7.1.4. Reagent water must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks. Reagent water must be shown to have a resistivity greater than or equal to 16.67 Mohm-cm.

7.2. Standards

7.2.1. All standards preparation, documentation and labeling must follow the requirements of STL-QA-0002.

7.2.2. Intermediate standards are purchased as custom multi-element mixes or as single-element solutions. All standards must be stored in FEP fluorocarbon or unused polyethylene or polypropylene bottles. Intermediate standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the intermediate solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem.

7.2.3. Working calibration and calibration verification solutions may be used for up to 1 month and must be replaced sooner if verification from an independent source indicates a problem. Standards should be prepared in a matrix of 5% hydrochloric and 5% nitric acids.

7.2.4. Refer to Tables III, IV, V and VI (Appendix A) for details regarding the working standard concentrations for calibration, calibration verification, interference correction and spiking solutions.

7.2.5. Initial calibration verification (ICV) standards are similar to calibration standards, but are from a completely different source.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Sample holding times for metals are six months from time of collection to the time of analysis.

8.2. Aqueous samples are preserved with nitric acid to a pH of <2 and may be stored in either plastic or glass. If boron or silica are to be determined, plastic containers are preferred. Refrigeration is not required. Preservation must be verified prior to analysis. For samples that will be analyzed by Method 200.7 for compliance with Safe Drinking Water regulations, the samples must be held for a minimum of 16 hours prior to verifying the pH.

8.3. Soil samples do not require preservation but must be stored at 4°C ± 2° until the time of preparation.

9. QUALITY CONTROL

9.1. QC requirements

9.1.1. Each analytical batch may contain up to 20 environmental samples, a method blank, and a single Laboratory Control Sample (LCS) and an MS/MSD pair. In the event that there is not sufficient sample to analyze an MS/MSD, an LCS duplicate (LCSD) is prepared and analyzed.

9.1.1.1. Unless authorized by QA, samples that have assigned QC limits different than the standard limits contained in QuantIMS QC code 01 must be batched separately, but can share the same QC samples.

9.1.1.2. Additional MS/MSDs do not count towards the 20 samples in an analytical batch.

9.1.1.3. A method blank must be included with each batch of samples. The matrix for aqueous and soil analyses is reagent (DI) water.

9.1.1.4. The LCS is spiked with all of the standard target compounds and is used to monitor the accuracy of the analytical process, independent of matrix effects. The matrix for aqueous analyses is reagent (DI) water and a standard reference material for solids.

9.1.1.5. All LCS and MS/MSD results – whether they pass criteria or not – are uploaded into the QuantIMS system for maintenance and periodic update of limits.

9.1.2. Instrument conditions must be the same for all standards, samples and QC samples.

9.1.3. All data will be reviewed by the analyst (1st review level) and then by a peer or supervisor (2nd level review).

9.2. Documentation

Table VII (Appendix A) provides a summary of quality control requirements including type, frequency, acceptance criteria and corrective action.

9.2.1. Initial Demonstration of Capability

Prior to analysis of any analyte using either Method 200.7 or Method 6010B, the following requirements must be met.

9.2.1.1. Instrument Detection Limit (IDL) - The IDL for each analyte must be determined for each analyte wavelength used on each instrument. The IDL must be determined annually at a minimum. If the instrument is adjusted in anyway that may affect the IDL, the IDL for that instrument must be redetermined. The IDL shall be determined by multiplying by 3, the average standard deviation obtained from the analysis of a standard solution (each analyte in reagent water) at a concentration 3x - 5x the previously determined IDL, with seven consecutive measurements on three nonconsecutive days. Each measurement must be performed as though it were a separate analytical sample (i.e., each measurement must be followed by a rinse and/or any other procedure performed between the analysis of separate samples). The result of the IDL determination must be below the reporting limit. The CLP IDL procedure can be used for this method.

9.2.1.2. Method Detection Limit (MDL) - An MDL must be determined for each analyte prior to the analysis of any client samples. The MDL is determined using seven replicates of reagent water, spiked with all the analytes of interest, that have been carried through the entire analytical procedure. MDLs must be redetermined on an annual basis in accordance with 40 CFR Part 136 Appendix B requirements as detailed in QA Policy QA-005. The spike level should be between the calculated MDL and 10x the MDL to be considered valid. In the event that the calculated MDL is less than one tenth the spike level the MDL data must be qualified and reviewed by the Quality Assurance manager. The result of the MDL determination must be below the reporting limit (RL). MDL studies for the determination of metals in soil need not be performed using spiked soil an appropriate soil MDL may be computed from the experimentally determined MDL for metals in aqueous solution once taken through the appropriate soil preparation procedure.

9.2.1.3. Linear Range Verification (LR) - The linear range must be determined on an annual basis for each analyte wavelength used on each instrument. The linear range is the concentration above which results cannot be reported without dilution of the sample. The standards used to define the linear range limit must be analyzed during a routine analytical run. For the initial determination of the upper limit of the linear dynamic range (LDR) for each wavelength, determine the signal responses from a minimum of three to five different concentration standards across the estimated range. One standard should be near the upper limit of the estimated range. The concentration measured at the LDR must be no more than 10% less than the expected level extrapolated from lower standards. If the instrument is adjusted in any way that may affect the LRs, new dynamic ranges must be determined. The LR data must be documented and kept on file. The quarterly CLP linear range methodology may be substituted for this requirement, using the 5% criteria.

9.2.1.4. Background Correction Points - To determine the appropriate location for off-line background correction when establishing methods, the user must scan the area on either side adjacent to the wavelength and record the apparent emission intensity from all other method analytes. This spectral information must be documented and kept on file. The location selected for background correction must be either free of off-line interelement spectral interference or a computer routine must be used for automatic correction on all determinations. Tests to determine spectral interference must be done using analyte concentrations that will adequately describe the interference. Background correction points must be set prior to determining IECs. Refer to the ICP instrument manual for specific procedures to be used in setting background correction points.

9.2.1.5. Inter-element Corrections (IECs) - ICP interelement correction factors must be determined prior to the analysis of samples and annually thereafter. If the instrument is adjusted in any way that may affect the IECs, the IECs must be redetermined. When initially determining IECs for an instrument, wavelength scans must be performed to ensure that solutions in use are free from contaminants. If an IEC varies significantly from the previously determined IEC then the possibility of contamination should be investigated. The purity of the IEC check solution can be verified by using a standard from a second source or an alternate method (i.e., GFAA or ICP-MS). Published wavelength tables (e.g. MIT tables, Inductively Coupled Plasma-Atomic Spectroscopy: Prominent Lines) can also be consulted to evaluate the validity of the IECs. Refer to the facility specific instrument operation SOP and instrument manufacturer's recommendations for specific procedures to be used in setting IECs. An IEC must be established to compensate for any interelement interference which results in a false analyte signal greater than $\pm$ the RL as defined in Tables I, IA or II. To determine IECs, run a single element standard at the

established linear range. To calculate an IEC, divide the observed concentration of the analyte by the observed concentration of the "interfering element."

Note: Trace ICP IECs are more sensitive to small changes in the plasma and instrument setup conditions. Adjustments in the IECs will be required on a more frequent basis for the Trace as reflected by the ICSA response.

9.2.1.6. Rinse Time Determination – If necessary, rinse times must be determined annually. To determine the appropriate rinse time for a particular ICP system, a linear range verification standard (see 9.2.1.3) should be aspirated as a regular sample followed by the analysis of a series of rinse blanks. The length of time required to reduce the analyte signals to < RL will define the rinse time for a particular ICP system. For some analytes it may be impractical to set the rinse time based on the linear range standard result (i.e., analyte not typically detected in environmental samples at that level and an excessive rinse time would be required at the linear range level). Until the required rinse time is established, the method recommends a rinse period of at least 60 seconds between samples and standards. If a memory effect is suspected, the sample must be reanalyzed after a rinse period of sufficient length. Rinse time studies can be conducted at additional concentration levels. These additional studies must be documented and kept on file, if a concentration other than the linear range level is used to set the rinse time. The concentration levels used to establish the rinse time must be taken into consideration when reviewing the data.

9.2.2. *Method Blank (MB) - One method blank must be processed with each preparation batch.* The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank should not contain any analyte of interest at or above the reporting limit (exception: common laboratory contaminants, see below) or at or above 5% of the measured concentration of that analyte in associated samples, whichever is higher (sample result must be a minimum of 10x higher than the blank contamination level).

For dissolved metals samples that have not been digested, a CCB result is reported as the method blank. The CCB run immediately prior to the start of the dissolved sample analyses must be used for this purpose. No more than 20 samples can be associated with one CCB.

9.2.3. Laboratory Control Sample (LCS) - One aqueous LCS must be processed with each preparation batch. The LCS must contain all analytes of interest and must be carried through the entire analytical procedure. Aqueous LCS spike levels are provided in Table III (Appendix A). The LCS is used to monitor the accuracy of the analytical process. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines. If the matrix is a solid, a soil LCS must be used.

For dissolved metals samples that have not been digested, a CCV result is reported as the LCS. The CCV run immediately prior to the start of the dissolved sample analyses must be used for this purpose. No more than 20 samples can be associated with one CCV.

9.2.4 Matrix Spike/Matrix Spike Duplicate (MS/MSD) - One MS/MSD pair must be processed for each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client specific data quality objectives (DQO's) may require the use of sample duplicates in place of or in addition to MS/MSDs. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked. Samples identified as field blanks cannot be used for MS/MSD analysis. Spiking levels are provided in Tables III and VI (Appendix A).

For dissolved metals samples which have not been digested, a MS/MSD must be performed per batch of up to 20 samples by spiking two aliquots of the sample at the levels specified in Table III (Appendix A).

9.2.5 *Dilution test* – A dilution test is performed to determine whether significant physical or chemical interferences exist due to the sample matrix. One sample per preparation batch must be processed as a dilution test. The test is performed by running a sample at a 5x (1:4) dilution. Samples identified as field blanks cannot be used for dilution tests. The results of the diluted sample, after correction for dilution, should agree within 10% of the original sample determination when the original sample concentration is greater than 50x the IDL. If the results are not within 10%, the possibility of chemical or physical interference exists.

9.2.6 Contract Required Detection Limit Test of the ICP (CRI)

Note: The CRI is a low-level CCV, sometimes referenced as the Low-Level Standard (LLS) of the analyte of interest. The CRA concentration is at or near the MDL and this QC parameter is not required for 200.7 or 6010B. The CRI is included for CLP work and upon client request.

Since the EPA has not set acceptance limits for the CRI the CRI will be controlled to the client requirements or ± 50%. A failure of this criteria requires that the system is evaluated and checked for error. A reanalysis is not absolutely necessary.

9.2.7 Initial Calibration Verification (ICV/ICB) - Calibration accuracy is verified by analyzing a second source standard (ICV). For analyses conducted under Method 200.7, the ICV result must fall within 5% of the true value for that solution with relative standard deviation <3% from replicate (minimum of two) exposures. For Method 6010B, the ICV must fall within 10% of the true value for that solution with relative standard deviation <5% from replicate (minimum of two) exposures. An ICB is analyzed immediately following the ICV to monitor low level accuracy and system cleanliness. The ICB result must fall within +/- the RL from zero. If either the ICV or ICB fail to meet criteria, the analysis should be terminated, the problem corrected, the instrument recalibrated and the calibration reverified. (See Section 11.11 for required run sequence).

9.2.8 Continuing Calibration Verification (CCV/CCB) - Calibration accuracy is monitored throughout the analytical run through the analysis of a known standard after every 10 samples. The CCV is a mid-range standard usually made from a dilution of the calibration standard. The CCV for both methods must fall within 10% of the true value for that solution with relative standard deviation <5% from replicate (minimum of two) exposures. A CCB is analyzed immediately following each CCV. (See Section 11.11 for required run sequence.) The CCB result must fall within +/- RL from zero. Sample results may only be reported when bracketed by valid CCV/CCB pairs. If a mid-run CCV or CCB fails, the analysis for the affected element must be terminated, the problem corrected, the instrument recalibrated, the calibration verified and the affected samples reanalyzed.

9.2.9 Interference Check Analysis (ICSA/ICSAB) - The validity of the interelement correction factors is demonstrated through the successful analysis of interference check solutions. The ICSA contains only interfering elements, the ICSAB contains analytes and interferents. Refer to Table V (Appendix A) for the details of ICSA and ICSAB composition. Custom multielement ICS solutions must be used. All analytes should be spiked into the ICSAB solution, therefore, if a non-routine analyte is required then it should be manually spiked into the ICSAB using a certified ultra high purity single element solution or custom lab-specific mix. If the ICP will display overcorrection as a negative number then the non-routine elements can be controlled from the ICSA. Elements known to be interferents on a required analyte must be included in the ICP run when that analyte is determined. Aluminum, iron, calcium and magnesium must always be included in all ICP runs.

9.2.9.1 The ICSA and ICSAB solutions must be run at the beginning and end of the run and every 8 hours. (See Section 11.11 for required run sequence.)

9.2.10 Method of Standard Addition (MSA) -This technique involves adding known amounts of standard to one or more aliquots of the processed sample solution. This technique compensates for a sample interferent that may enhance or depress the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences which cause a baseline shift. Refer to Appendix D for specific MSA requirements.

9.2.11 Quality Assurance/Project Summaries - Certain clients may require project- or program-specific QC which may supersede this SOP requirements. Quality Assurance Summaries (QASs) or equivalent documents providing project-specific requirements should be developed so that project staff clearly understand the special project requirements.

9.3 Procedural Variations

9.3.4 One time procedural variations are allowed only if deemed necessary in the professional judgment of the analyst to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

9.3.5 Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

9.4 Nonconformance and Corrective Action

Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the facility QA Manager.

9.5 QC Program

Further details of QC and corrective action guidelines are presented in the QC Program policy document (QA-003).

10 CALIBRATION AND STANDARDIZATION

10.2 Set up the instrument with the operating parameters recommended by the manufacturer. Allow the instrument to become thermally stable before beginning calibration (approximately 30 minutes of warm-up is required).

10.3 Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures. Flush the system with the calibration blank between each standard or as the manufacturer recommends. The calibration curve must consist of a minimum of a blank and a standard. Refer to the ICP instrument manual for a detailed set up and operation protocols.

10.4 Calibration must be performed daily and each time the instrument is set up. Instrument runs may be continued over periods exceeding 24 hours as long as all calibration verification (CCV) and interference check QC criteria are met. The instrument standardization date and time must be included in the raw data.

10.5 Refer to Appendix H for DOE requirements.

10.6 If the calibration curve does not meet method requirements,

10.6.4.1 Evaluate whether the problem is related to the analytical range. The low standard or the high standard having a response that is out of line with the others typically expresses this. In such situations, the analyst should determine whether the analytical range should be decreased by deleting the high or low standard. If the low standard is deleted, adjust the reporting limit. If the high standard is deleted, dilute samples appropriately. If the high standard shows saturation of the response, the calibration range must be adjusted.

10.6.4.2 The minimum number of calibration points for DOE analyses must be four points with a coefficient of determination (R^2) for all regression curve being equal to or greater than 0.995.

11 PROCEDURE

11.2 For 200.7 analyses, dissolved (preserved) samples must be digested unless it can be documented that the sample meets all of the following criteria:

A. Visibly transparent with a turbidity measurement of 1 NTU or less.

B. Is of one liquid phase and free of particulate or suspended matter following acidification.

C. Is NOT being analyzed for silver.

11.3 A minimum of two exposures for each standard, field sample and QC sample is required. The average of the exposures is reported. For Trace ICP analyses, the results of the sum channel must be used for reporting.

11.4 Prior to calibration and between each sample/standard the system is rinsed with the calibration blank solution. The minimum rinse time between analytical samples is 60 seconds unless following the protocol outlined in 9.2.1.6 it can be demonstrated that a shorter rinse time may be used. Triton-X can be added to the rinse solution to facilitate the rinse process.

11.5 The use of an autosampler for all runs is strongly recommended.

11.6 The use of automated QC checks through the instrument software is highly recommended for all calibration verification samples (ICV, CCV), blanks (ICB, CCB, PB), interference checks (ICSA, ICSAB) and field samples (linear range) to improve the data review process.

11.7 To facilitate the early identification of QC failures and samples requiring rerun it is strongly recommended that sample data is reviewed periodically throughout the run.

11.8 To facilitate the data review and reporting processes it is strongly recommended that all necessary dilutions be performed before closing out the instrument run.

11.9 For unattended overnight auto-runs it is strongly recommended that the frequency of ICSA/ICSAB analysis be increased to every 4 hours.

11.10 The use of an internal standard is recommended on the conventional, non-Trace ICPs as an alternative to using the method of standard additions. This technique is useful in overcoming matrix interferences especially in high solids matrices. However, for conventional ICP techniques, internal standards may not be necessary provided that one of the following is performed to minimize physical interferences: (1) peristaltic pump is used, (2) high solids nebulizer is used, or (3) high solids samples are diluted and reanalyzed.

11.11 The use of an internal standard is **required** on the Trace ICP unless a high solids nebulizer and mass flow controller is utilized or the calibration and QC standards are matrix matched. Normal acid concentrations are 5% HCl and 5% HNO3

The following procedural guidelines must be followed when using an internal standard:

11.11.4 Typically used internal standards are: yttrium or scandium. (Note: Any element can be used that is not typically found in environmental samples at a high rate of occurrence.)

11.11.5 The internal standard (IS) must be added to every sample and standard at the same concentration. It is recommended that the IS be added to each analytical sample automatically through use of a third pump channel and mixing coil. Internal standards should be added to blanks, samples and standards in a like manner, so that dilution effects resulting from the addition may be disregarded.

11.11.6 The concentration of the internal standard should be sufficiently high to obtain good precision in the measurement of the IS analyte used for data correction and to minimize the possibility of correction errors if the IS analyte is naturally present in the sample.

11.11.7 The internal standard raw intensity counts must be printed on the raw data.

11.11.8 The analyst must monitor the response of the internal standard throughout the sample analysis run. This information is used to detect potential problems and identify possible background contributions from the sample (i.e., natural occurrence of IS analyte).

11.11.8.1 If the internal standard counts fall within ±30% of the counts observed in the ICB then the data is acceptable.

11.11.8.2 If the internal standard counts in the field samples are more than ±30% higher than the expected level, the field samples must then be:

1. Diluted and reanalyzed;
2. The IS concentrations must be raised; or
3. A different internal standard must be used.

11.12 The following analytical sequence must be used for Methods 6010B and 200.7:

Instrument Calibration
ICV
ICB
CRI*
ICSA
ICSAB
8 samples (7 samples if CRI is run)
CCV
CCB
10 samples
CCV
CCB
Repeat sequence of up to 8 samples between CCV/CCB pairs as required to complete run
ICSA
ICSAB
CCV
CCB

* The CRI will be added if required.

Refer to Quality Control Section 9.0 and Table VII (Appendix A) for Method 6010B and 200.7 quality control criteria.

11.13 Additional quality control analyses are necessary for analysis under the Contract Laboratory Program (CLP). If these are included then CLP, 6010 and 200.7 samples can be included in the same sequence. Refer to CORP-MT-002 for details.

11.14 Full method required QC must be available for each wavelength used in determining reported analyte results.

11.15 Guidelines are provided in the appendices on procedures to minimize contamination of samples and standards, preventive maintenance and troubleshooting.

11.16 All measurements must fall within the defined linear range where spectral interference correction factors are valid. Dilute and reanalyze all samples for required analytes that exceed the linear range or use an alternate wavelength for which QC data are established. If an interelement correction exists for an analyte which exceeds the linear range, the IEC may be inaccurately applied. Normally the inability to accurately correct for an interference is denoted by a "K" on the instrument printout. Therefore, even if an overrange analyte may not be required to be reported for a sample, if that analyte is a interferent for any requested analyte in that sample, the sample must be diluted. Acid strength must be maintained in the dilution of samples.

11.17 For TCLP samples, full four-point MSA will be required if all of the following conditions are met:

1) recovery of the analyte in the matrix spike is not at least 50%,
2) the concentration of the analyte does not exceed the regulatory level, and,
3) the concentration of the analyte is within 20% of the regulatory level.

The reporting and regulatory limits for TCLP analyses as well as matrix spike levels are detailed in Table VI (Appendix A). Appendix D provides guidance on performing MSA analyses.

11.18 Any variation in procedure shall be completely documented using instrument run logs, maintenance logs, report narratives, a Nonconformance Memo, or an anomaly report and is approved by a Supervisor/Group Leader and QA Manager. If contractually required, the client shall be notified by the Project Manager.

11.19 Nonconformance documentation shall be filed in the project file.

11.20 Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

12 DATA ANALYSIS AND CALCULATIONS

12.2 Raw data report in mg/L concentration off the curve.

12.3 ICV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(ICV)}{True(ICV)}\right)$$

12.4 CCV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(CCV)}{True(CCV)}\right)$$

12.5 Matrix Spike Recoveries are calculated according to the following equation:

$$\%R = 100\left(\frac{SSR - SR}{SA}\right)$$

Where:
SSR = Spike Sample Result
SR = Sample Result
SA = Spike Added

12.6 The relative percent difference (RPD) of matrix spike/matrix spike duplicates are calculated according to the following equations:

$$RPD = 100\left[\frac{|MSD - MS|}{\left(\frac{MSD + MS}{2}\right)}\right]$$

Where:
MS = determined spiked sample concentration
MSD = determined matrix spike duplicate concentration

12.7 The final concentration for a digested aqueous sample is calculated as follows:

$$mg/L = \frac{C \times V1 \times D}{V2}$$

Where:
C = Concentration (mg/L) from instrument readout
D = Instrument dilution factor
V1 = Final volume in liters after sample preparation
V2 = Initial volume of sample digested in liters

12.8 The final concentration determined in digested solid samples when reported on a dry weight basis is calculated as follows:

$$mg/Kg, dry\ weight = \frac{C \times V \times D}{W \times S}$$

Where:

C = Concentration (mg/L) from instrument readout
D = Instrument dilution factor
V = Final volume in liters after sample preparation
W = Weight in Kg of wet sample digested
S = Percent solids/100

Note: A Percent Solids determination must be performed on a separate aliquot when dry weight concentrations are to be reported. If the results are to be reported on wet weight basis the "S" factor should be omitted from the above equation.

12.9 The LCS percent recovery is calculated according to the following equation:

$$\%R = 100\left(\frac{Found(LCS)}{True(LCS)}\right)$$

12.10 The dilution test percent difference for each component is calculated as follows:

$$\%Difference = \frac{|I - S|}{I} \times 100$$

Where:

I = Sample result (Instrument reading)
S = Dilution test result (Instrument reading × 5)

12.11 Appropriate factors must be applied to sample values if dilutions are performed.

12.12 Sample results should be reported with up to three significant figures in accordance with the significant figure policy.

13. DATA ASSESSMENT AND ACCEPTANCE CRITERIA

The following represent data assessment for samples and acceptance criteria for QC measures. Corrective actions for any failure in QC measures are shown in Section 14.

12.13 QC sample acceptance criteria

12.13.4 Method Blank.

12.13.4.1 No target analytes may be present in the method blank above the reporting limit with the exception of the common laboratory contaminates (Pb, Zn, Cu, and Fe).

12.13.5 Laboratory Control Sample (LCS).

12.13.5.1 Until in-house control limits are established, method 6010B will have a control limit of 80 – 120% recovery will be applied. For method 200.7, 6010B a control limit of 85 – 115% recovery will be applied.

12.13.5.2 The RPD for a LCS and LCSD will be compared to the MS/MSD RPD requirement of 20%.

12.13.5.3 All control analytes must be within established control limits for accuracy (%Recovery) and precision (RPD). Exceptions are allowed only with QA and project management approval.

12.13.5.4 It is desirable to have all non-control analytes within control limits, but it is expected that a small proportion will not be in control.

12.13.6 Matrix Spike/Matrix Spike Duplicate (MS/MSD).

12.13.6.1 Until in-house control limits are established, methods 6010B and 200.7 will have a control limit of 75 – 125% recovery will be applied for the MS/MSD.

12.13.6.2 The RPD for a MS/MSD pair is 20% (35% for soil).

12.13.6.3 All analytes should be within established control limits for accuracy (%Recovery) and precision (RPD). Deviations from this may be the results of matrix effects, which are confirmed by passing LCSs.

12.13.6.4 No specific corrective actions are required in the evaluation of the MS/MSD results provided that the batch LCS is in control. Analysts should use sound judgment in accepting MS/MSD results that are not within control limits, especially if the LCS results are borderline.

12.13.7 Low-Level Contract Required Detection Limit Test (CRI)

12.13.7.1 Since the EPA has not set acceptance limits for the CRI the CRI will be controlled to the client requirements or ± 25%.

12.13.8 Initial Calibration Verifications (ICV).

12.13.8.1 For analyses conducted under Method 200.7, the ICV result must fall within 5% of the true value for that solution with relative standard deviation <3% from replicate (minimum of two) exposures. For Method 6010B, the ICV must fall within 10% of the true value for that solution with relative standard deviation <5% from replicate (minimum of two) exposures. If the ICV or fails to meet criteria, the analysis should be terminated, the problem corrected, the instrument recalibrated and the calibration reverified. (See Section 11.11 for required run sequence).

12.13.9 Initial Calibration Blank (ICB).

12.13.9.1 An ICB is analyzed immediately following the ICV to monitor low level accuracy and system cleanliness. The ICB result must fall within +/- the RL from zero. If the ICB fails to meet criteria, the analysis should be terminated, the problem corrected, the instrument recalibrated and the calibration reverified. (See Section 11.11 for required run sequence).

12.13.10 Continuing Calibration Verifications (CCV)

12.13.10.1 The CCV for both methods must fall within 10% of the true value for that solution with relative standard deviation <5% from replicate (minimum of two) exposures. A CCB is analyzed immediately following each CCV. (See Section 11.11 for required run sequence.) The CCB result must fall within +/- RL from zero. Sample results may only be reported when bracketed by valid CCV/CCB pairs. If a mid-run CCV, the analysis for the affected element must be terminated, the problem corrected, the instrument recalibrated, the calibration verified and the affected samples reanalyzed.

12.13.11 Continuing Calibration Blank (CCB)

12.13.11.1 The CCB result must fall within +/- RL from zero. Sample results may only be reported when bracketed by valid CCV/CCB pairs. If a mid-run CCB fails, the analysis for the affected element must be terminated, the problem corrected, the instrument recalibrated, the calibration verified and the affected samples reanalyzed.

12.13.12 Interference Check Analysis (ICSA/ICSAB)

12.13.12.1 The ICSAB results for interferents must fall within 80 – 120% of the true value.

12.13.12.2 ICSA results for the non-interfering elements with RLs < 10 µg/L must fall within ± 2x RL from zero. ICSA results for the non-interfering elements with RLs > 10 µg/L must fall within ± 1xRL from zero.

12.14 Sample result evaluation

12.14.4 Carryover. When a sample has a high response for a compound, there is a real possibility that some of the sample may carry over into the sample analyzed immediately afterward.

12.14.4.1 If a sample analyzed after a sample with high concentrations has negative results, carryover did not occur.

12.14.4.2 If a sample analyzed after a sample with high concentrations has positive results for the same analytes, or if the chromatographic profile resembles the previous sample, the results

are questionable. This sample must be reanalyzed under conditions in which carryover can be confirmed to not have occurred.

12.14.5 Dilutions

12.14.5.1 If the response for any compound exceeds the working range of the analytical system, a dilution of the extract is prepared and analyzed. An appropriate dilution should be in the upper half of the calibration range.

12.14.5.2 If the initial diluted run has no hits or hits below 20% of the calibration range and the matrix allows for analysis at a lesser dilution, then the sample must be reanalyzed at a dilution targeted to bring the largest hit in the upper half of the calibration range.

13 CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

13.2 Method Blank.

13.2.4 The samples in the batch associated to the defective method blank are evaluated:

13.2.4.1 If the analyte found in the method blank is confirmed to not be present in any of the associated samples at any level, the contamination did not affect those samples. This represents a non-impact situation and no specific corrective action is taken. A nonconformance memo is written to notify project management of the situation for evaluation against project requirements.

13.2.4.2 If the analyte is a common laboratory contaminant (copper, iron, lead (Trace only) or zinc) the data may be reported with qualifiers if the concentration of the analyte in the method blank is less than two times the RL. **Such action must be taken in consultation with the client and must be addressed with a Non Conformance Memorandum, (NCM) in the project narrative.**

13.2.4.3 If the analyte found in the method blank is confirmed to be present in one or more of the associated samples, the concentrations are compared.

13.2.4.3.1 Repreparation and reanalysis of all samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples (see exception noted in 14.1.1.2).

13.2.4.3.2 If there is no analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. **Such action must be addressed in an NCM.**

13.2.4.4 If the criteria of 14.1.1.3 are not met and reanalysis is not possible, then the sample data must be qualified. **This anomaly must be addressed with a NCM in the project narrative and the client must be notified.**

13.2.4.5 If the analyte found in the method blank is not one of the common laboratory contaminants (Fe, Pb [Trace only], Cu, and Zn) and routine corrective actions do not return the system to service, the analytical system must be placed out of service until the source of the problem is identified and corrected.

13.3 Laboratory Control Sample

13.3.4 If any control analyte is out of control for accuracy (% Recovery), the associated samples are evaluated.

13.3.4.1 If the recovery is biased high [>120% for 6010 and >115% for 200.7] and the associated samples have results <RL for that analyte, a non-impact situation ensues. A nonconformance memo is written to notify project management of the situation for evaluation against project requirements. **Based upon client consultation, the samples may have to be rerun.**

13.3.4.2 If the recovery is biased low and the associated samples have positive results for that analyte, a minimal impact situation ensues. A nonconformance memo is written to notify

project management of the situation for evaluation against project requirements. **Based upon client consultation, the samples (and batch) may have to be rerun.**

13.3.4.3 If the recovery is biased high and the associated samples have positive results for that analyte, the prescribed corrective action is redigestion of the samples and reanalysis of the batch.

13.3.4.4 If the recovery is biased low and the samples have no positive results for that analyte, the prescribed corrective action is redigestion of the samples and reanalysis of the batch.

13.3.5 If any control analyte is out of control for precision (RPD), the associated samples are evaluated. All decisions about corrective action(s) are made in consultation with the project manager.

13.3.5.1 If there are no positive results in one or more samples, a non-impact situation ensues for those samples. A nonconformance memo is written to notify project management of the situation for evaluation against project requirements.

13.3.5.2 If there are positive results for one or more analytes, the likelihood of poor reproducibility increases and corrective action must be evaluated. A nonconformance memo is written to notify project management of the situation for a project decision on whether the affected sample(s) should be reanalyzed.

13.3.6 In the event that an MS/MSD analysis is not possible, a Laboratory Control Sample Duplicate (LCSD) must be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits

13.4 Matrix Spike/Matrix Spike Duplicate

13.4.4 If any control analyte is out of control for accuracy (% Recovery), the associated samples are evaluated.

13.4.4.1 If any analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. For both methods 200.7 and 6010B, control limits of 75 - 125% recovery and 20% RPD or historical acceptance criteria must be applied to the MS/MSD. If the LCS recovery is within limits, then the laboratory operation is in control and the results may be accepted. If the recovery of the LCS is outside limits corrective action must be taken. Corrective action will include repreparation and reanalysis of the batch. MS/MSD results which fall outside the control limits must be addressed in the narrative.

13.4.4.2 If the native analyte concentration in the MS/MSD exceeds 4x the spike level for that analyte, the recovery data are reported as NC (i.e., not calculated). If the reporting software does not have the ability to report NC then the actual recovery must be reported and narrated as follows: "Results outside of limits do not necessarily reflect poor method performance in the matrix due to high analyte concentrations in the sample relative to the spike level."

13.4.4.3 If an MS/MSD is not possible due to limited sample volume then a laboratory control sample duplicate (LCSD) should be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.

13.5 Low-Level Contract Required Detection Limit Test (CRI)

13.5.4 If the CRI fails the system will be evaluated and checked for errors. A failure of the CRI does not necessitate reanalysis.

13.6 Initial Calibration Verification (ICV)

13.6.4 If the ICV or fails to meet criteria [±5% for Method 200.7 or ±10% for method 6010B], the analysis should be terminated, the problem corrected, the instrument recalibrated and the calibration reverified. (See Section 11.11 for required run sequence).

13.7 Initial Calibration Blank (ICB)

13.7.4 If the ICB or fails to meet criteria [within +/- the RL from zero], the analysis should be terminated, the problem corrected, the instrument recalibrated and the calibration reverified.

13.8 Continuing Calibration Verification (CCV)

13.8.4 The CCV for both methods must fall within 10% of the true value for that solution with relative standard deviation <5% from replicate (minimum of two) exposures. Sample results may only be reported when bracketed by valid CCV/CCB pairs. If a mid-run CCV, the analysis for the affected element must be terminated, the problem corrected, the instrument recalibrated, the calibration verified and the affected samples reanalyzed.

13.9 Continuing Calibration Blank (CCB)

13.9.4 The CCB result must fall within +/- RL from zero. Sample results may only be reported when bracketed by valid CCV/CCB pairs. If a mid-run CCB fails, the analysis for the affected element must be terminated, the problem corrected, the instrument recalibrated, the calibration verified and the affected samples reanalyzed.

13.10 Interference Check Analysis (ICSA/ICSAB)

13.10.4 If any results for the interferents for the ICSA/ICSAB are out of control, the associated samples are evaluated.

13.10.4.1 If any ICSAB interferent results fail criteria, the analysis should be terminated, the problem corrected, the instrument recalibrated and the samples rerun.

13.10.4.2 If the ICSA results for the non-interfering elements do not fall within +/- 2x RL (RL ≤10) or ± 1xRL (RL>10) from zero the field sample data must be evaluated as follows:

13.10.4.2.1 If the non-interfering element concentration in the ICSA is the result of contamination versus a spectral interference, and this reason is documented, the field sample data can be accepted.

13.10.4.2.2 If the affected element was not required then the sample data can be accepted.

13.10.4.2.3 If the interfering elements are not present in the field sample at a concentration which would result in a false positive or negative result greater than +/- 2x RL from zero then the field sample data can be accepted.

13.10.4.2.4 If the interfering element is present in the field sample at a level which would result in a false analyte signal greater than ± 2x RL from zero, the data can be accepted only if the concentration of the affected analyte in the field sample is more than 10x the analyte signal in the ICSA.

13.10.4.2.5 If the data does not meet the above conditions then the IECs must be re-evaluated and corrected if necessary and the affected samples reanalyzed or the sample results manually corrected through application of the new IEC to the raw results. If the results are recalculated manually the calculations must be clearly documented on the raw data.

14 CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

14.2 Method blanks

14.2.4 If there is insufficient sample to perform re-analysis, the analyst must notify the project manager for consultation with the client. In this situation, all associated samples are flagged with a "B" or appropriate qualifier and appropriate comments in the narrative.

14.3 LCS

14.3.4 If the batch is not redigested and reanalyzed, the reasons for accepting the batch must be clearly presented in the project records and the report. Acceptable matrix spike recoveries are not sufficient justification to preclude re-extraction of the batch.

14.3.5 If re-extraction and reanalysis of the batch is not possible due to limited sample volume or other constraints, the LCS is reported, all associated samples are flagged, and appropriate comments are made in a narrative to provide further documentation.

14.4 Insufficient sample

14.4.4 If there is insufficient sample to repeat the analysis, the project manager is notified via NCM for consultation with the client.

15 METHOD PERFORMANCE

15.2 Each laboratory must have initial demonstration of performance data on file for each analyte of interest as described in Section 9.0.

15.3 Refer to Tables I, IA & II in Appendix A for the list of Method 6010B and 200.7 analytes as well as additional analytes that may be analyzed using this SOP.

15.4 Method performance is determined by the analysis of MS and MSD samples as well as method blanks and laboratory control samples. The MS or MSD recovery should fall within +/- 25 % and the MS/MSD should compare within 20% RPD or within the laboratory's historical acceptance limits. These criteria apply to analyte concentrations greater than or equal to 10xIDL. Method blanks must meet the criteria specified in Section 9.2.2. The laboratory control samples should recover within 20% (15% for 200.7) of the true value or within the laboratory's historical acceptance limits.

15.5 Training Qualification:

The group/team leader or the supervisor has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

16 POLLUTION PREVENTION

This procedure will be carried out in a manner consistent with all applicable federal state and local regulations regarding pollution control and prevention. Specific controls due to the accidental release of hazardous materials can be found in the Chemical Hygiene Plan and facility attachments.

17 WASTE MANAGEMENT

17.2 Waste generated in this procedure will be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Coordinator should be contacted if additional information is required.

17.3 Standards should be purchased and prepared in volumes consistent with laboratory use to minimize the volume of expired standards to be disposed.

18 REFERENCES

18.2 40 CFR Part 136, Appendix B, 7-5-95, Determination of Method Detection Limits.

18.3 Test Methods for Evaluating Solid Waste , Physical/Chemical Methods, SW-846, 3rd Edition, Final Update III, Revision 2, December 1996. Method 6010B.

18.4 Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry, Revision 4.4, May 1994. Method 200.7.

18.5 CORP-MT-0002, Inductively Coupled Plasma-Atomic Emission Spectroscopy, Method 200.7 & CLP-M, SOW ILMO3.0.

19 CLARIFICATIONS, MODIFICATIONS AND ADDITIONS TO REFERENCE METHOD

19.2 Modifications/Interpretations from reference method

19.2.4 Modifications/interpretations from both Methods 6010B and 200.7.

19.2.4.1 STL laboratories use mixed calibration standard solutions purchased from approved vendors instead of using individual mixes prepared in house as recommended by the subject methods.

19.2.4.2 The alternate run sequence presented in Section 11.11 is consistent with method requirements. Additional QC (i.e., ICSA) analyses were added to accommodate the 200.7 protocol requirements.

19.2.4.3 Methods 200.7 and 6010B state that if the correction routine is operating properly, the determined apparent analyte(s) concentration from analysis of each interference solution should fall within a specific concentration range around the calibration blank. In determining IECs, because of lack of definition clarification for "concentration range around the calibration blank," STL has adopted the procedure in EPA CLP ILMO3.0.

19.2.4.4 Section 8.5 of Method 6010B and Section 9.5 of Method 200.7 recommend that whenever a new or unusual matrix is encountered, a series of tests be performed prior to reporting concentration data for that analyte. The dilution test helps determine if a chemical or physical interference exists. Because STL laboratories receive no prior information from clients regarding when to expect a new or unusual matrix, STL may select to perform a dilution test on one sample in each prep batch. According to the method, the post digestion spike (PDS) determines any potential matrix interferences. At STL labs, matrix interference is determined by evaluating data for the LCS and MS/MSD. STL requires documented, clear guidance when a new or unusual matrix will be received for a project and a request to perform the dilution test or PDS on a client-identified sample.

19.2.4.5 A low-level CCV (CRI) has been has been incorporated into the run sequence.

19.2.5 Modifications from Method 200.7.

19.2.5.1 Method 200.7 defines the IDL as the concentration equivalent to a signal, due to the analyte, which is equal to three times the standard deviation of a series of ten replicate measurements of the calibration blank signal at the same wavelength. STL labs utilize the CLP IDL definition as defined in Section 9.2.1.1 of this SOP.

19.2.5.2 The calibration blank is prepared in an acid matrix of 5% HNO_3/5% HCl instead of the specified 2% HNO_3/10% HCl matrix as the former matrix provides for improved performance relative to the wide variety of digestate acid matrices which result from the various EPA preparation protocols applied.

19.2.5.3 Method section 9.3.4 specifies that "Analysis of the IPC (ICSA/AB) solution immediately following calibration must verify that the instrument is within ± 5% of calibration with a relative standard deviation <3% from replicate integrations ≥ 4." STL uses a minimum of two exposures.

19.2.5.4 Section 7.12 of 200.7 indicates that the QCS (ICV) should be prepared at a concentration near 1 ppm. The ICV specified in this SOP deviates from the 1 ppm criteria. For the analytes, this SOP specifies ICV concentrations which are appropriate to the range of calibration. The intent of the ICV, verification of calibration standard accuracy, is independent of the ICV concentration used.

19.2.5.5 The ICS criteria applied by this SOP differ from those stated in the method. Method 200.7 section 10.4 states that results should fall within the established control limits of 3 times the standard deviation of the calibration blank for that analyte. The control limits listed in this SOP are those applicable to the EPA designed solution.

19.2.5.6 Method 200.7 section 9.3.4 states the CCB should be less than the IDL, but > the lower 3-sigma control limit of the calibration blank. The intent of this requirement is to ensure that the calibration is not drifting at the low end. STL has adopted an absolute control limit of +/- RL from zero for calibration blank criteria. SOP section 9.7 provides the detailed corrective action criteria that must be followed.

19.2.6 Modifications from Method 6010B.

19.2.6.1 Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the MDL. This SOP states that the method blank must not contain any analyte of interest at or above the reporting limit. Common lab contaminants are allowed up to two times the reporting limit in the blank following consultation with the client.

19.2.6.2 Method 6010B section 8.6.1.3 states that the results of the calibration blank are to agree within 3x the IDLIf not, repeat the analysis two or more times and average the results. If the average is not within three standard deviation of the background mean, terminate the analysis, correct the problem, recalibrate, and reanalyze the previous 10 samples. The intent of this requirement is to ensure that the calibration is not drifting at the low end. STL has adopted an absolute control limit of +/- RL from zero for calibration blank criteria. See SOP Section 9.2.7 for a detailed description of the required corrective action procedures.

19.3 Modifications from previous SOP

Refer to revision 2.1 of this SOP.

19.4 Facility-Specific SOPs

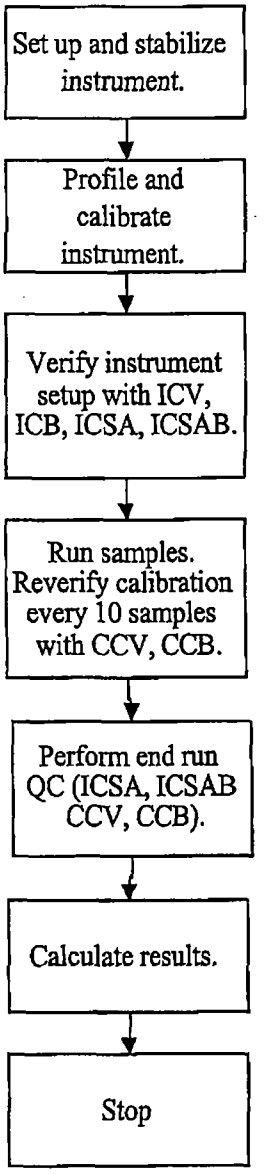
This SOP has been modified to depict the standard operating procedures at STL-St. Louis.

19.5 Documentation and Record Management

The following documentation comprises a complete ICP raw data package:

- Raw data (direct instrument printout).
- Relevant sample preparation benchsheets.
- Run log printout from instrument software where this option is available (TJA) or manually generated run log (i.e., Ward WSL printout).
- Data review checklist - See Appendix B.
- Standards documentation (including prep and expiration dates, source, and lot #).
- Nonconformance/anomaly documentation (if applicable).

20.5. Flow Diagram



APPENDIX A

TABLES

TABLE I. Method 200.7 and 6010B Target Analyte List

ELEMENT	Symbol	CAS #	6010B analyte	200.7 analyte	Reporting Limit (ug/L) Water	Reporting Limit (mg/kg) Soil
Aluminum	Al	7429-90-5	X	X	200	20
Antimony	Sb	7440-36-0	X	X	60	6
Arsenic	As	7440-38-2	X	X	300	30
Barium	Ba	7440-39-3	X	X	200	20
Beryllium	Be	7440-41-7	X	X	5.0	0.5
Boron	B	7440-42-8		X	200	20
Cadmium	Cd	7440-43-9	X	X	5.0	0.5
Calcium	Ca	7440-70-2	X	X	5000	500
Chromium	Cr	7440-47-3	X	X	10	1
Cobalt	Co	7440-48-4	X	X	50	5
Copper	Cu	7440-50-8	X	X	25	2.5
Iron	Fe	7439-89-6	X	X	100	10
Lead	Pb	7439-92-1	X	X	100	10
Lithium	Li	7439-93-2	X	X	50	5
Magnesium	Mg	7439-95-4	X	X	5000	500
Manganese	Mn	7439-96-5	X	X	15	1.5
Molybdenum	Mo	7439-98-7	X	X	40	4
Nickel	Ni	7440-02-0	X	X	40	4
Phosphorus	P	7723-14-0	X	X	300	30
Potassium	K	7440-09-7	X	X	5000	500
Selenium	Se	7782-49-2	X	X	250	25
Silicon	Si	7631-86-9		X	500	N/A
Silver	Ag	7440-22-4	X	X	10	1
Sodium	Na	7440-23-5	X	X	5000	500
Strontium	Sr	7440-24-6	X		50	5
Thallium	Tl	7440-28-0	X	X	2000	200
Vanadium	V	7440-62-2	X	X	50	5
Zinc	Zn	7440-66-6	X	X	20	2

TABLE IA. Method 200.7 and 6010B Trace ICP Target Analyte List

ELEMENT	Symbol	CAS #	Reporting Limit (ug/L) Water	Reporting Limit (mg/kg) Soil
Arsenic	As	7440-38-2	10	1.0
Lead	Pb	7439-92-1	3.0	0.3
Selenium	Se	7782-49-2	5.0	0.5
Thallium	Tl	7440-28-0	10	1.0
Antimony*	Sb	7440-36-0	10	1.0
Cadmium*	Cd	7440-43-9	2.0	0.2
Silver*	Ag	7440-22-4	5.0	0.5
Chromium*	Cr	7440-47-3	5.0	0.5

* Note: Reporting limits for indicated elements are reported at the levels listed in Table I. When client specifications require a lower reporting limit Table IA limits may be used, provided suitable MDL determinations are available.

TABLE II. Non-Routine Analyte List

ELEMENT	Symbol	CAS #	Reporting Limit (ug/L) Water	Reporting Limit (mg/kg) Soil
Tin	Sn	7440-31-5	100	10
Titanium	Ti	7440-32-6	50	5
Bismuth	Bi	7440-06-99	200	20
Zirconium	Zr	7440-06-77	100	10
Tungsten	W	7440-03-37	500	50
Tellurium	Te	1349-48-09	500	50
Thorium	Th	7440-02-91	500	50
Uranium	U	7440-06-11	500	50
Palladium	Pd	7440-00-53	100	10

NOTE: Analysis of all elements listed may not be available at all STL facilities.

TABLE III. Matrix Spike and Aqueous Laboratory Control Sample Levels

ELEMENT	LCS Level (ug/L)	Matrix Spike Level (ug/L)
Aluminum	1000	2000
Antimony	1000	500
Arsenic	1000	2000
Barium	1000	2000
Beryllium	1000	50
Cadmium	1000	50
Calcium	20000	50000
Chromium	1000	200
Cobalt	1000	500
Copper	1000	250
Iron	1000	1000
Lead	1000	500
Lithium	200	2000
Magnesium	20000	50000
Manganese	1000	500
Molybdenum	1000	1000
Nickel	1000	500
Phosphorous	10000	10000
Potassium	20000	50000
Selenium	1000	2000
Silver	250	50
Sodium	20000	50000
Strontium	1000	1000
Thallium	1000	2000
Vanadium	1000	500
Zinc	1000	500
Boron	2000	2000
Silicon	2000	10000
Tin	2000	2000
Titanium	2000	1000
Bismuth	2000	2000
Zirconium	2000	2000
Tellurium	2000	2000
Thorium	2000	2000
Uranium	2000	2000

TABLE IV. ICP Calibration and Calibration Verification Standards

Element	Calibration Level	RL (ug/L)	ICV (ug/L)	CCV (ug/L)
Aluminum	100000	200	50000	40000
Antimony	10000	60	5000	4000
Arsenic	10000	300(10)	5000	4000
Barium	10000	200	5000	4000
Beryllium	10000	5	5000	4000
Cadmium	10000	5	5000	4000
Calcium	50000	5000	25000	20000
Chromium	10000	10	5000	4000
Cobalt	10000	50	5000	4000
Copper	10000	25	5000	4000
Iron	100000	100	50000	40000
Lead	10000	100(3)	1000	4000
Lithium	10000	50	1000	4000
Magnesium	50000	5000	25000	20000
Manganese	10000	15	5000	4000
Molybdenum	10000	40	5000	4000
Nickel	10000	40	5000	4000
Phosphorous	10000	300	5000	4000
Potassium	100000	5000	50000	40000
Selenium	10000	250(5)	5000	4000
Silver	2000	10	1250	1000
Sodium	100000	5000	50000	40000
Strontium	10000	50	5000	4000
Thallium	10000	2000(10)	5000	4000
Vanadium	10000	50	5000	4000
Zinc	10000	20	5000	4000
Boron	10000	200	5000	4000
Silicon	10000	500	5000	4000
Tin	10000	100	5000	4000
Titanium	10000	50	5000	4000
Bismuth	10000	200	5000	4000
Zirconium	10000	100	5000	4000
Tellurium	10000	500	5000	4000
Thorium	10000	500	5000	4000
Uranium	10000	500	5000	4000

TABLE V. Interference Check Sample Concentrations*

Element	ICSA (ug/L)	ICSAB (ug/L)
Aluminum	500000	500000
Antimony	-	1000
Arsenic	-	1000
Barium	-	500
Beryllium	-	500
Cadmium	-	1000
Calcium	500000	500000
Chromium	-	500
Cobalt	-	500
Copper	-	500
Iron	200000	200000
Lead	-	1000
Magnesium	500000	500000
Manganese	-	500
Molybdenum	-	1000
Nickel	-	1000
Potassium	-	10000
Selenium	-	1000
Silver	-	1000
Sodium	-	10000
Thallium	-	1000**
Vanadium	-	500
Zinc	-	1000
Tin	-	1000

* Custom solutions contain analytes common to all STL facilities. Non-routine elements not listed above should be spiked into the ICSAB at 1000 ug/L.

** Thallium level for standard ICP should be at 10000 ug/L.

TABLE VI. TCLP Reporting Limits, Regulatory Limits and Matrix Spike Levels

ELEMENT	Reporting Level* (ug/L)	Regulatory Limit (ug/L)	Spike Level (ug/L)
Arsenic	300	5000	5000
Barium	200	100000	50000
Cadmium	50	1000	1000
Chromium	100	5000	5000
Lead	100	5000	5000
Selenium	50	1000	1000
Silver	100	5000	1000

*Note: In cases of limited sample volume or matrix interference's in which dilutions are required trace reporting limits will be used to quantify results for TCLP extracts to ensure regulatory compliance.

TABLE VII. Summary Of Quality Control Requirements

QC PARAMETER	FREQUENCY*	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Two-point Initial Calibration	Beginning of every analytical run, every 24 hours, whenever instrument is modified, or CCV criterion is not met	RSD between duplicate exposures ≤5%	Terminate analysis; Correct the problem; Prepare new standards; Recalibrate following system performance.
ICV	Beginning of every analytical run.	Method 200.7: 95 – 105 % recovery. Method 6010B: 90 – 110 % recovery.	Terminate analysis; Correct the problem; Recalibrate.
ICB	Beginning of every analytical run, immediately following the ICV.	The result must be within +/- RL from zero.	Terminate analysis; Correct the problem; Recalibrate.
LLCCV (CRI)	1 per run	+ 25% or as required by contract	Evaluate the system for error, or as required by contract.
CCV	Every 10 samples and at the end of the run.	Method 200.7 & 6010B: 90 – 110 % recovery.	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCV.
CCB	Immediately following each CCV.	The result must be within +/- RL from zero.	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCB.
ICSA	Beginning of every run	See Section 9.2.8.3	See Section 9.2.8.3.
ICSAB	Immediately following each ICSA.	Results must be within 80 - 120% recovery.	See Section 9.2.8.2

* See Sections 11.11 for exact run sequence to be followed.

TABLE VII. Summary of Quality Control Requirements (Continued)

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Dilution Test	One per prep batch.	For samples > 50x IDL, dilutions must agree within 10%.	Narrate the possibility of physical or chemical interference per client request.
Method Blank	One per sample preparation batch of up to 20 samples.	The result must be less than or equal to the RL. Common lab contaminants may be accepted up to 2x the RL after consultation with the client (See 9.2.2). Sample results greater than 10x the blank concentration are acceptable. Samples for which the contaminant is < RL may not require redigestion or reanalysis (see Section 9.2.2).	Redigest and reanalyze samples. Note exceptions under criteria section. See Section 9.2.2 for additional requirements.
Laboratory Control Sample (LCS)	One per sample preparation batch of up to 20 samples.	Aqueous LCS must be within 80 - 120% recovery or in-house control limits. (85-115% for 200.7) Samples for which the contaminant is < RL and the LCS results are > 120% (115% for 200.7) may not require redigestion or reanalysis (see Section 9.2.3)	Terminate analysis; Correct the problem; Redigest and reanalyze all samples associated with the LCS.

TABLE VII. Summary of Quality Control Requirements (Continued)

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Matrix Spike	One per sample preparation batch of up to 20 samples.	75 - 125 % recovery or in-house control limits. If for TCLP See Section 11.16.	In the absence of client specific requirements, flag the data; no flag required if the sample level is > 4x the spike added. For TCLP see Section 11.16.
Matrix Spike Duplicate	See Matrix Spike	75 - 125 % recovery; RPD ≤ 20% .	See Corrective Action for Matrix Spike.

APPENDIX B

ICP DATA REVIEW CHECKLIST

ICP Data Review Checklist

Run/Project Information:

Run Date: _____ **Analyst:** _____ **Instrument:** _____
Prep Batches Run: _____

Circle Methods used: 6010B / 200.7: CORP-MT-0001STL Rev 2.1
CLP ILMO3.0/4.0 : CORP-MT-0002STL Rev 1.1

Review Items

A. Calibration/Instrument Run QC	Yes	No	N/A	2nd Level
1. Instrument calibrated per manufacturer's instructions and at SOP specified levels ?				
2. ICV/CCV analyzed at appropriate frequency and within control limits ? (6010B, CLP = 90 - 110%, 200.7 = 95 -105%[ICV])				
3. ICB/CCB analyzed at appropriate frequency and within +/- RL or +/- CRDL (CLP) ?				
4. CRI analyzed? (for CLP only)				
5. ICSA/ICSAB run at required frequency and within SOP limits ?				
B. Sample Results				
1. Were samples with concentrations > the linear range for any parameter diluted and reanalyzed ?				
2. All reported results bracketed by in control QC ?				
3. Sample analyses done within holding time ?				
C. Preparation/Matrix QC				
1. LCS done per prep batch and within QC limits ?				
2. Method blank done per prep batch and < RL or CRDL (CLP) ?				
3. MS run at required frequency and within limits ?				
4. MSD or DU run at required frequency and RPD within SOP limits ?				
5. Dilution Test done per prep batch (or per SDG for CLP) ?				
6. Post digest spike analyzed if required (CLP only) ?				
D. Other				
1. Are all nonconformances documented appropriately ?				
2. Current IDL/LR/IEC data on file ?				
3. Calculations checked for error ?				
4. Transcriptions checked for error ?				
5. All client/project specific requirements met ?				
6. Date/time of analysis verified as correct ?				

Analyst: _____ **Date:** _____
Comments: _____

2nd Level Reviewer : _____ **Date:** _____
Comments: _____

APPENDIX C

CROSS REFERENCE OF TERMS USED IN METHODS 6010B, 200.7 AND STL SOP

CROSS REFERENCE OF TERMS COMMONLY USED IN METHODS EPA 200.7, SW6010B, AND STL SOP

EPA 200.7	SW6010B	STL SOP
Calibration blank (CB)	Calibration blank	Initial and continuing calibration blanks (ICB/CCB)
Dilution test	Dilution test	Dilution Test
Instrument detection limit (IDL)	Instrument detection limit (IDL)	Instrument detection limit (IDL)
Instrument performance check (IPC)	Continuing calibration verification (CCV)	Continuing calibration verification (CCV)
Internal standard	Internal standard	Internal standard (IS)
Laboratory duplicates	n/a	n/a
Laboratory fortified blank (LFB)	n/a	Laboratory control sample (LCS)
Laboratory fortified sample matrix (LFM)	Matrix spike and matrix spike duplicate (MS/MSD)	Matrix spike and matrix spike duplicate (MS/MSD)
Laboratory reagent blank (LRB)	Method blank	Method or Prep blank (MB)
Linear dynamic range (LDR)	Linear dynamic range (LDR)	Linear dynamic range (LDR)
Method detection limit (MDL)	Method detection limit (MDL)	Method detection limit (MDL)
Quality control sample (QCS)	Check standard or Initial calibration verification (ICV)	Initial calibration verification (ICV)
Spectral interference check solution (SIC)	Interference check solution (ICS)	Interference check solution (ICSA/ICSAB)

APPENDIX D

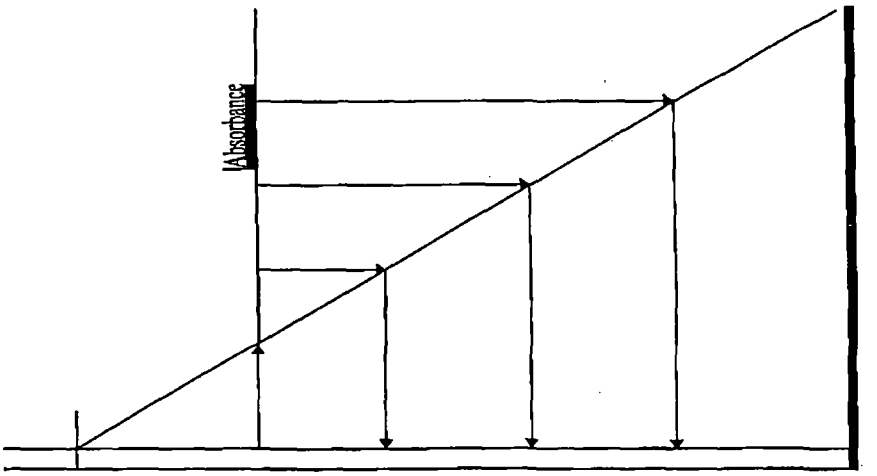
MSA GUIDANCE

Appendix D. MSA Guidance

Method of Standard Addition

Four equal volume aliquots of sample are measured and known amounts of standards are added to three aliquots. The fourth aliquot is the unknown and no standard is added to it. The concentration of standard added to the first aliquot should be 50% of the expected concentration. The concentration of standard added to the second aliquot should be 100% of the expected concentration and the concentration of standard added to the third aliquot should be 150% of the expected concentration. The volume of the unspiked and spiked standard should be the same.

In order to determine the concentration of analyte in the sample, the analytical value of each solution is determined and a plot or linear regression performed. On the vertical axis the analytical value is plotted versus the concentrations of the standards on the horizontal axis. An example plot is shown in Figure 1. When the resulting line is extrapolated back to zero absorbance, the point of interception of the horizontal axis is the concentration of the unknown.



For the method of standard additions to be correctly applied, the following limitations must be taken into consideration:

- The plot of the sample and standards must be linear over the concentration range of concern. For best results, the slope of the curve should be similar to that of a plot of the aqueous standard curve.
- The effect of the interference should not vary as the ratio of the standard added to the sample matrix changes.

APPENDIX E

TROUBLESHOOTING GUIDE

APPENDIX E. TROUBLESHOOTING GUIDE

Problem	Possible Cause/ Solution
High Blanks	Increase rinse time Clean or replace tip Clean or replace torch Clean or replace sample tubing Clean or replace nebulizer Clean or replace mixing chamber Lower Torch
Instrument Drift	RF not cooling properly Vacuum level is too low Replace torch (Crack) Clean or replace nebulizer (blockage) Check room temperature (changing) Replace pump tubing Room humidity too high Clean torch tip (salt buildup) Check for argon leaks Adjust sample carrier gas Reprofile Horizontal Mirror Replace PA tube
Erratic Readings, Flickering Torch or High RSD	Check for argon leaks Adjust sample carrier gas Replace tubing (clogged) Check drainage(back pressure changing) Increase uptake time (too short) Increase flush time (too short) Clean nebulizer, torch or spray chamber Increase sample volume introduced Check that autosampler tubes are full Sample or dilution of sample not mixed Increase integration time (too short) Realign torch Reduce amount of tubing connectors
Cu/Mn Ratio Outside Limits or Low Sensitivity	Plasma conditions changed Clean nebulizer, torch or spray chamber Replace tubing (clogged) Realign torch Check IECs
Standards reading twice normal absorbance or concentration	Incorrect standard used Incorrect dilution performed

APPENDIX F

CONTAMINATION CONTROL GUIDELINES

APPENDIX F. CONTAMINATION CONTROL GUIDELINES

The following procedures are strongly recommended to prevent contamination:

All work areas used to prepare standards and spikes should be cleaned before and after each use.

All glassware should be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water.

Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.

Powdered or Latex Gloves must not be used in the metals laboratory since the powder contains silica and zinc as well as other metallic analytes. Only vinyl or nitrile gloves should be used in the metals laboratory.

Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.

Autosampler trays should be covered to reduce the possibility of contamination. Trace levels of elements being analyzed in the samples can be easily contaminated by dust particles in the laboratory.

The following are helpful hints in the identification of the source of contaminants:

Yellow pipet tips and volumetric caps can sometimes contain cadmium.

Some sample cups have been found to contain lead.

The markings on glass beakers have been found to contain lead. If acid baths are in use for glassware cleaning, they should be periodically checked for contaminants since contaminant concentrations will increase over time.

New glassware especially beakers can be a source of silica and boron.

Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.

Improper cleaning of glassware can cause contamination.

Latex gloves contain over 500 ppb of zinc.

APPENDIX G

PREVENTIVE MAINTENANCE

APPENDIX G. PREVENTIVE MAINTENANCE

A maintenance log is used to record when maintenance is performed on instruments. When an instrument problem occurs indicate the date, time and instrument number, then identify the problem and corrective action in the maintenance log.

The following procedures are required to ensure that that the instrument is fully operational.

Daily	Change sample pump tubing and pump windings Check argon gas supply level Check rinse solution and fill if needed Check waste containers and empty if needed Check sample capillary tubing is clean and in good condition Check droplet size to verify nebulizer is not clogged. Check sample flow for cross flow nebulizer Check sensitivity ratio-should be 30% of value at date that IECs were performed Check pressure for vacuum systems
As Needed	Clean plasma torch assembly to remove accumulated deposits Clean nebulizer and drain chamber; keep free-flowing to maintain optimum performance Replace peristaltic pump tubing, sample capillary tubing and autosampler sipper probe Apply silicon spray on autosampler tracks
Weekly	Check water level in coolflow
Monthly	Clean air filters on back of power unit to remove dust
Yearly	Check oil for vacuum systems Replace coolant water filter (may require more or less frequently depending on quality of cooling water)

APPENDIX H

DOE PROCEDURAL REQUIREMENTS

APPENDIX H: DOE PROCEDURAL REQUIREMENTS

Scope and Application

To better document the required calibration procedures for DOE-AL for trace ICP analysis the following procedural attachment has been prepared.

Summary

For the trace analysis of arsenic, lead, selenium, thallium and antimony a multi-point calibration curve is prepared daily. The TJA61e is then calibrated using the "DOE" calibration method. This method uses a zero and four additional calibration points to calibrate the previously mentioned elements.

Standard Levels

The calibration and check standard levels are prepared from a variety of concentrated stock solutions including the normal calibration and calibration verification standards. In addition to the normal stock standards, a standard specially purchased is also used. The table below illustrates the final concentrations of the various standards.

Standard Levels, ug/L

Element	S Low	S Med	S High	CAL1	ICVDOE	CCVDOE
Arsenic	100	500	1000	10000	500	400
Lead	100	500	1000	10000	500	400
Selenium	100	500	1000	10000	500	400
Thallium	100	500	1000	10000	500	400
Antimony	100	500	1000	10000	500	400

Quality Control

In addition to the standard QA/QC requirements as defined by the method and SOP, the correlation coefficient, R, is required to be 0.995 or greater for each of the reported elements using a multi-point calibration. A low level CR1 and spike is required for the elements listed above.

Attachment QAPP-C3

Preparation and Analysis of Mercury in Solid Samples by Cold Vapor Atomic Absorption Spectroscopy

Method 245.5 CLP-M, SOW ILM03.0

SOP No.:	CORP-MT-0008STL
Revision No.:	0.2
Revision Date:	8/5/00
Page:	1 of 30
Implementation Date:	04/24/01

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TITLE: PREPARATION AND ANALYSIS OF MERCURY IN SOLID SAMPLES BY COLD VAPOR ATOMIC ABSORPTION SPECTROSCOPY, METHOD 245.5 CLP-M, SOW ILM03.0

(SUPERSEDES: CORP-MT-0008, REVISION 0.1)

Prepared by: _____

Approved by: _____
Technical Specialist

Approved by: _____
Quality Assurance Manager

Approved by: _____
Environmental Health and Safety Coordinator

Approved by: _____
Laboratory Director

Proprietary Information Statement:

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1. SCOPE AND APPLICATION

1.1. This procedure describes the preparation and analysis of mercury (Hg, CAS # 7439-97-6) by Cold Vapor Atomic Absorption Spectroscopy (CVAA) using Method 245.5 CLP-M as contained in CLP SOW ILMO3.0. In addition to SOW ILMO3.0, this SOP is also compliant with the requirements of CLP SOWs 7/88, 3/90, ILMO1.0 and ILMO2.1.

1.2. CVAA analysis provides for the determination of total mercury (organic and inorganic). The combination of the oxidants, potassium permanganate and potassium persulfate, has been found to give 100% recovery with both types of compounds. Detection limits, sensitivity and optimum concentration ranges for mercury analysis will vary with the matrices, instrumentation and volume of sample used.

1.3. Methods 245.5 CLP-M is applicable to the preparation and analysis of mercury in soils, sediments, bottom deposits and sludge-type materials. All matrices require sample preparation prior to analysis.

1.4. The CRDL for mercury in solid matrices is 0.1 mg/kg based a 0.2 g sample aliquot (wet weight).

1.5. Method detection limits are maintained in the Information Management System (QuantIMS). Because of their dynamic nature, they are not specifically listed in this document, but can be retrieved at any time using TraQAr tools.

1.6. Quality control limits (accuracy and precision for spikes) are also maintained in QuantIMS, and are also dynamic. Therefore, they are not specifically listed in this document, but can be retrieved at any time using TraQAr tools.

1.7. Additional compounds may be amenable to this method. The minimum requirement for non-standard analytes is that the reporting limit be set at the lowest required concentration that can actually be detected by the instrument, and when an MDL study can not be conducted, the MDL be set equal to the reporting limit.

2. SUMMARY OF METHOD

2.1. This SOP describes a technique for the determination of mercury in solution. The procedure is a physical method based on the absorption of radiation at 253.7 nm by mercury vapor. A representative portion of the sample is digested in sulfuric and nitric acids. Organic mercury compounds are oxidized with potassium permanganate and potassium persulfate and the mercury reduced to its elemental state with stannous chloride and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance is measured as a function of mercury concentration. Concentration of the analyte in the sample is determined by comparison of the sample absorbance to the calibration curve (absorbance vs. concentration).

3. DEFINITIONS

3.1. See policy QA-003 for definitions

3.2. Total Metals - the concentration determined on an unfiltered sample following digestion.

4. INTERFERENCES

Chemical and physical interferences may be encountered when analyzing samples using this method.

4.1. Potassium permanganate which is used to breakdown organic mercury compounds also eliminates possible interferences from sulfide. Concentrations as high as 20 mg/L of sulfide as sodium sulfide do not interfere with the recovery of inorganic mercury from reagent water.

4.2. Copper has also been reported to interfere; however, copper concentrations as high as 10 mg/L had no effect on the recovery of mercury from spiked samples.

4.3. Chlorides can cause a positive interference. Samples high in chlorides require additional permanganate (as much as 25 mL) because, during the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation at 253.7 nm. Care must be taken to ensure that free chlorine is absent before the mercury is reduced and

swept into the cell. This is accomplished by adding excess hydroxylamine reagent (25 mL). Both inorganic and organic mercury spikes have been quantitatively recovered from seawater using this technique.

Note: Sufficient addition of permanganate is apparent when the purple color persists at least 15 minutes. Some samples may require dilution prior to digestion due to extremely high concentrations of chloride.

4.4. Interference from certain volatile organic materials that absorb at this wavelength may also occur. If suspected, a preliminary run without stannous chloride can determine if this type of interference is present. While the possibility of absorption from certain organic substances present in the sample does exist, this problem is not routinely encountered. This is mentioned only to caution the analyst of the possibility. If this condition is found to exist, the mercury concentration in the sample can be determined by subtracting the result of the sample run without the reducing reagent (stannous chloride) from that obtained with the reducing reagent.

4.5. Samples containing high concentrations of oxidizable organic materials, as evidenced by high COD levels, may not be completely oxidized by this procedure. When this occurs the recovery of mercury will be low. The problem can be eliminated by reducing the volume of original sample used.

4.6. The most common interference is laboratory contamination which may arise from impure reagents, dirty glassware, improper sample transfers, dirty work areas, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.

5. SAFETY

5.1. Procedures shall be carried out in a manner that protects the health and safety of all associates.

5.2. The Chemical Hygiene Plan (CHP) gives details about the specific health and safety practices which are to be followed in the laboratory area. Personnel must receive training in the CHP, including the written Hazard Communication plan, prior to working in the laboratory. Consult the CHP, the Health and Safety Policies and Procedures Manual, and available Material Safety Data Sheets (MSDS) prior to using the chemicals in the method.

5.3. Consult the Health and Safety Policies and Procedures Manual for information on Personal Protective Equipment. Eye protection that satisfies ANSI Z87.1 (as per the Chemical Hygiene Plan), laboratory coat and appropriate gloves must be worn while samples, standards, solvents and reagents are being handled. Disposable gloves that have become contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.4. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the Material Safety Data Sheets (MSDS) maintained in the laboratory. The following specific hazards are known:

5.4.1. The following materials are known to be **corrosive**:

Hydrochloric acid, nitric acid and sulfuric acid

5.4.2. The following materials are known to be **oxidizing agents**:

Nitric acid, potassium permanganate, potassium persulfate and magnesium perchlorate

5.4.3. Mercury is a highly toxic element that must be handled with care. The analyst must be aware of the handling and clean-up techniques before working with mercury. Since mercury vapor is toxic, precaution must be taken to avoid its inhalation, ingestion or absorption through skin. All lines should be checked for leakage and the mercury vapor must be vented into a hood or passed through a mercury absorbing media such as:

5.4.3.1. Equal volumes of 0.1 M $KMnO_4$ and 10% H_2SO_4, or

5.4.3.2. Iodine, 0.25%, in a 3% KI solution.

5.4.4. Magnesium sulfate is known to be a reproductive toxin (mutagen).

5.5. Exposure to chemicals will be maintained **as low as reasonably achievable,** therefore, unless they are known to be non-hazardous, all samples will be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.6. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operations will permit.

5.7. All work must be stopped in the event of a known, or potential compromise to the health or safety of any associate. The situation must be reported **immediately** to a laboratory supervisor.

5.8. Do not look directly into the beam of the Hg lamp. The UV light that these lamps radiate is harmful to the eyes.

5.9. Cylinders of compressed gas must be handled with caution, in accordance with local regulations. It is recommended that, wherever possible, cylinders be located outside the laboratory and the gas led to the instrument through approved lines.

5.10. The CVAA apparatus must be properly vented to remove potentially harmful fumes generated during sample analysis.

5.11. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices outlined in the Health and Safety Manual. These employees must have training on the hazardous waste disposal practices initially upon assignment of these tasks, followed by an annual refresher training.

6. EQUIPMENT AND SUPPLIES

6.1. Temperature controlled water bath (capable of maintaining a temperature of 90-95 °C).

6.2. Leeman Labs Mercury Analyzer

6.3. 50 ml container or equivalent.

6.4. Nitrogen or argon gas supply, welding grade or equivalent.

6.5. Calibrated automatic pipettes or Class A glass volumetric pipettes.

6.6. Class A volumetric flasks.

6.7. Top-loading balance, capable of reading up to two decimal places.

6.8. Thermometer (capable of accurate readings at 95 °C).

6.9. Disposable cups or tubes.

7. REAGENTS AND STANDARDS

7.1. Reagents

7.1.1. All reagent preparation is documented in the electronic standards log. All reagents are labeled with their unique ID, name (and concentration, if applicable) of the reagent, the date prepared and the expiration date.

7.1.2. Reagent water must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

7.1.3. Nitric acid (HNO_3), concentrated, trace metal grade or better.

Note: If a high reagent blank is obtained, it may be necessary to distill the nitric acid.

7.1.4. Sulfuric acid (H_2SO_4), concentrated, trace metal grade or better.

7.1.5. Stannous sulfate solution: Add 25 g of stannous sulfate to 250 mL of 0.5 N sulfuric acid. This mixture is a suspension and should appear cloudy. This solution should be made daily and should be stirred continuously during use.

Note: Stannous chloride may be used in place of stannous sulfate. Prepare the stannous chloride solution according to the recommendations provided by the instrument manufacturer. Dissolve 50g of stannous chloride into 500 ml of 10% HCl.

7.1.6. Sodium chloride-hydroxylamine hydrochloride solution: Add 12 g of sodium chloride and 12 g of hydroxylamine hydrochloride to every 100 mL of reagent water or a certified stock standard may be purchased from a vendor.

Note: Hydroxylamine sulfate may be used in place of hydroxylamine hydrochloride.

7.1.7. Potassium permanganate, 5% solution (w/v): Dissolve 5 g of potassium permanganate for every 100 mL of reagent water or a certified stock reagent may be purchased from a vendor.

7.1.8. Potassium persulfate, 5% solution (w/v): Dissolve 5 g of potassium persulfate for every 100 mL of reagent water or a certified stock reagent may be purchased from a vendor.

7.2. Standards

7.2.1. All standards preparation, documentation and labeling must follow the requirements of SOP STL-QA-0002.

7.2.2. Stock (100 ppm) mercury standards are purchased as custom solutions. All standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the stock solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem.

7.2.3. Intermediate mercury standard (0.1 ppm): Take 0.1 mL of the stock mercury standard (7.2.2) and dilute to 100 mL with reagent water. The intermediate standard must be made daily and must be prepared in a matrix of 2% HNO_3. This acid (2 mL of concentrated HNO_3) must be added to the flask/bottle before the addition of the stock standard aliquot.

7.2.4. Intermediate mercury QC standard (0.1 ppm): Must be prepared from a different stock (different lot) than that of the Intermediate mercury standard. Take 0.1 mL of the stock mercury standard (7.2.2) and dilute to 100 mL with reagent water. The intermediate standard must be made daily and must be prepared in a matrix of 2% HNO_3. This acid (2 mL of concentrated HNO_3) must be added to the flask/bottle before the addition of the stock standard aliquot.

7.2.5. The calibration standards must be prepared fresh daily from the working standard by transferring 0, 0.2, 0.5, 1.0, 5.0 and 10.0 mL aliquots of the working mercury standard into sample prep bottles and proceeding as specified in Section 11.1

Note: Alternate volumes of standard may be prepared as long as the accuracy and final standard concentrations as detailed in Table I are maintained.

7.2.6. The initial calibration verification standard must be made from a different stock solution than that of the calibration standards.

7.2.7. Solid reference material for use as soil LCS (see 7.2.7)

7.2.8. For analyses performed under direct contract to the USEPA CLP, the EPA provided ICV solution and solid LCS material must be used.

7.2.9. Refer to Table I (Appendix A) for details regarding the working standard concentrations for calibration, calibration verification and spiking solutions. All standards must be processed through the entire analytical procedure including sample preparation.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. The sample holding time for mercury is 26 days from time of receipt to the time of analysis.

8.2. Soil samples do not require preservation but must be stored at 4° C ± 2° C until the time of analysis.

9. QUALITY CONTROL

9.1. QC requirements

9.1.1. Each analytical batch may contain up to 20 environmental samples, a method blank, and a single Laboratory Control Sample (LCS) and an MS/MSD pair. In the event that there is not sufficient sample to analyze an MS/MSD, an LCS duplicate (LCSD) is prepared and analyzed.

9.1.1.1. Samples that have assigned QC limits different than the standard limits contained in QuantIMS QC code 01 must be batched separately, but can share the same QC samples.

9.1.1.2. Additional MS/MSDs do not count towards the 20 samples in an analytical batch.

9.1.1.3. A method blank must be included with each batch of samples.

9.1.1.4. The LCS is spiked with all of the standard target compounds and is used to monitor the accuracy of the analytical process, independent of matrix effects.

9.1.1.5. All LCS and MS/MSD results – whether they pass criteria or not – are uploaded into 's QuantIMS system for maintenance and periodic update of limits.

9.1.2. Instrument conditions must be the same for all standards, samples and QC samples.

9.1.3. All data will be reviewed by the analyst (1st review level) and then by a peer or supervisor (2nd level review).

9.2. Documentation

The CLP SOW ILMO3.0 document provides further details of the QC and corrective action guidelines presented in this SOP. Refer to this document if additional guidance is required

Table II (Appendix A) provides a summary of quality control requirements including type, frequency, acceptance criteria and corrective action.

9.2.1. Initial Demonstration of Capability

Prior to the analysis of any analyte using 245.5 CLP-M, the following requirements must be met.

9.2.1.1. Instrument Detection Limit (IDL) -The IDL for each analyte must be determined for each analyte wavelength used on each instrument. The IDL must be determined quarterly (every three months). If the instrument is adjusted in anyway that may affect the IDL, the IDL for that instrument must be redetermined. For example, the IDL must be redetermined after the instrument is moved or the internal optics cleaned. The IDL shall be determined by multiplying by 3, the average of the standard deviations obtained on three nonconsecutive days from the analysis of a standard solution (analyte in reagent water) at a concentration 3 - 5 times the previously determined IDL, with seven consecutive measurements per day. When initially determining the IDL for a new instrument, use the manufacturer's IDL as a reference point for making up the 3 - 5x IDL standard. Each measurement must be performed as though it were a separate analytical sample (i.e., each measurement must be followed by a rinse and/or any other

procedure performed between the analysis of separate samples). The result of the IDL determination must be below the CRDL.

- If the IDLs are not completed by the quarterly due date, no CLP analyses may be performed until the IDLs are completed (IDLs can not be retroactively applied).
- For reporting purposes, if multiple CVAA instruments are used for the analysis of an element in a SDG, the highest CVAA IDL must be used for reporting concentration values.
- Instrument specific IDLs must be used for all calculations.
- When calculating IDLs always round the IDL up and to the appropriate number of significant figures (one decimal place).

9.2.1.2. Sample Delivery Group (SDG)- An SDG is defined in the CLP SOW by the following, whichever is more frequent:

- each Case of field samples received, OR
- each 20 field samples within a Case, OR
- each 14 day calendar day period during which field samples in a Case are received.

For commercial clients, the SDG definition must be clarified with the client prior to the analysis of samples.

9.2.2. Analytical Sample - The CLP SOW defines the term "analytical sample" to include all field samples and all required QC samples (matrix spikes, duplicates, postdigestion spikes, serial dilutions, laboratory control samples, interference check samples, CRDL standards, preparation blanks and linear range analyses) except those directly related to instrument calibration or calibration verification (calibration standards, ICV/ICB and CCV/CCB).

9.2.3. Preparation Batch- The CLP SOW defines batch as "A group of samples prepared at the same time in the same location using the same method".

9.2.4. Preparation Blank (PB) - One preparation blank (method blank) must be processed with each preparation batch (or with each SDG, whichever is more frequent for U.S.EPA CLP contract analyses). The preparation blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The preparation blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The preparation blank should not contain any analyte of interest at or above the CRDL (exception: see below).

- If any analyte concentration in the blank is above the CRDL, the lowest concentration of that analyte in the associated samples must be 10X the blank concentration. Otherwise, all samples associated with the blank, with the analyte's concentration less than 10X the blank concentration and above the CRDL, must be redigested and reanalyzed for that analyte (except for identified aqueous soil field blanks).
- If the concentration of the blank is below the negative CRDL, then all samples reported below 10X CRDL associated with the blank must be redigested and reanalyzed.
- If the above criteria are not met and reanalysis is not possible, then the sample data must be qualified. **This anomaly must be addressed in the project narrative and the client must be notified.**

9.2.5. Laboratory Control Sample (LCS) - One solid LCS must be processed with each preparation batch (or with each SDG, whichever is more frequent for U.S.EPA CLP contract analyses). The LCS is used to monitor the accuracy of the analytical process. The solid LCS control limits shall be those established by the supplier of the solid LCS. Corrective action, when LCS results fail to meet control limits, will be repreparation and reanalysis of the batch.

9.2.6. Matrix Spike (MS)- One matrix spike must be processed for each SDG or each group of samples of a similar matrix type, whichever is more frequent. A matrix spike is a field sample to which known concentrations of target analytes have been added prior to digestion. Samples identified as field blanks cannot be used for matrix spike analysis. The matrix spike results are used to determine the effect of a matrix on the accuracy of the analytical process. Spiking levels are provided in Table I.

- If any analyte recovery falls outside the control limits of 75-125%, the data of all samples associated with that spiked sample must be flagged appropriately. An exception to the rule is granted if the native analyte concentration in the MS exceeds 4x the spike level for that analyte. In such an event, the data shall be reported unflagged even if the recovery does not meet the criteria.
- If the MS recovery falls outside the control limits and the sample result does not exceed 4x the spike added, the data must be appropriately flagged.
- The average of the duplicate results cannot be used for the purpose of determining percent recovery.

9.2.7. Duplicate (DU)- One duplicate must be processed for each SDG or each group of samples of a similar matrix type, whichever is more frequent. Samples identified as field blanks cannot be used for duplicate sample analysis.

- If the sample results are greater than or equal to five times the CRDL, a control limit of ± 20 % RPD must be used.
- If either the sample or sample duplicate value is less than five times the CRDL, a control limit of ± the CRDL must be used.
- If both the sample and the sample duplicate are below the IDL, the RPD is not calculated and no control criteria apply.
- If the DU RPD is outside control limits, the data is flagged appropriately.

9.2.8. Initial Calibration Verification (ICV/ICB) - Calibration accuracy is verified by analyzing a second source standard (ICV) immediately after calibration. The ICV result must fall within 20% of the true value. An ICB is analyzed immediately following the ICV to monitor low level accuracy and system cleanliness. The ICB result must fall within +/- the CRDL from zero. If either the ICV or ICB fail to meet criteria, the analysis should be terminated, the problem corrected and the instrument recalibrated. (See Section 11.2.10 for required run sequence). If the cause of the ICV or ICB failure was not directly instrument related the corrective action will include repreparation of the associated samples.

9.2.9. CRDL Standard (CRA) - To verify linearity near the CRDL for CVAA analysis, a CRA standard is run at the beginning of each sample analysis run, but not before the ICV. The CRA standard must contain the analyte at the CRDL. (See Section 11.2.10 for required run sequence.)

9.2.10. Continuing Calibration Verification (CCV/CCB) - Calibration accuracy is monitored throughout the analytical run through the analysis of a known standard after every 10 samples or every 2 hours, whichever is more frequent. The CCV must be a mid-range standard at a concentration other than that of the ICV. The CCV result must fall within 20% of the true value for that solution. A CCB is analyzed immediately following each CCV. (See Section 11.2.10 for required run sequence.) The CCB result must fall within +/- CRDL from zero. If a mid-run CCV or CCB fails, the analysis must be terminated, the problem corrected, the instrument recalibrated, the calibration verified and the affected samples reanalyzed. If the cause of the CCV or CCB failure was not directly instrument related, the corrective action will include repreparation of the associated samples.

- Each CCV analyzed must reflect the conditions of analysis of all associated samples. The duration of analysis, rinses, and other operations that may affect the CCV result may not differ from those used for the associated samples. For example, the difference in time between a CCV analysis and the CCB immediately following it or the sample immediately preceding it may not exceed the smallest difference between any two consecutive samples associated with that CCV.

9.2.11. Quality Assurance Summaries - Certain clients may require specific project or program QC which may supersede the SOP requirements. Quality Assurance Summaries (QAS) should be developed to address these requirements.

9.3. Procedural Variations

9.3.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of the analyst to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

9.3.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

9.4. Nonconformance and Corrective Action

Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the facility QA Manager.

9.5. QC Program

Further details of QC and corrective action guidelines are presented in the QC Program policy document (QA-003).

10. CALIBRATION AND STANDARDIZATION

10.1. Initial Calibration

10.1.1. Calibration standards must be processed through the preparation procedure as described in Section 11.1.

10.1.2. Due to the differences in preparation protocols separate calibration and calibration verification standards must be prepared for aqueous and solid matrices.

10.1.3. Calibration must be performed daily (every 24 hours) and each time the instrument is set up. The instrument calibration date and time must be included in the raw data.

10.1.4. Set up the instrument with the operating parameters recommended by the manufacturer. Allow the instrument to become thermally stable before beginning calibration (approximately 30 minutes of warm-up is required).

10.1.5. Calibrate the instrument according to instrument manufacturer's instructions, using a minimum of five standards and a blank. One standard must be at the reporting limit. Analyze standards in ascending order beginning with the blank. Refer to Section 7.2.4 and Table I for additional information on preparing calibration standards and calibration levels.

10.1.6. The calibration curve must have a correlation coefficient of ≥ 0.995 or the instrument shall be stopped and recalibrated prior to running samples. Sample results can not be reported from a curve with an unacceptable correlation coefficient.

10.1.7. If the calibration curve does not meet method requirements,

10.1.7.1. Evaluate whether the problem is related to the analytical range. The low standard or the high standard having a response that is out of line with the others typically expresses this. In such situations, the analyst should determine whether the analytical range should be decreased by deleting the high or low standard. If the low standard is deleted, adjust the reporting limit. If the high standard is deleted, dilute samples appropriately. If the high standard shows saturation of the response, the calibration range must be adjusted. |

10.1.7.2. Evaluate whether the problem was related to a misinjection of one of the standards. This must be confirmed by reanalyzing the suspect standard. If the reanalyzed standard is in line with the others, the misinjection of the original standard is confirmed. At this point, the analyst has the choice of deleting the offending calibration point, or replacing it with the new run. The minimum number of calibration points must be five.

10.1.7.3. The relationship between the standard mass and the response in the calibration standards can be fitted to a linear or quadratic regression curve. In all cases, the coefficient of determination (R^2) for all regression curves must be equal to or greater than 0.990.

10.2. Initial calibration verification

10.2.1. Refer to Section 9.0 for calibration verification procedures, acceptance criteria and corrective actions.

10.2.2. Not meeting this requirement may be indicative of serious system malfunction or inaccuracies in the standards used for the initial calibration curve or ICV standard. Corrective action must be taken (including reanalysis of the ICV, or analysis of a different ICV). Any decision to proceed with analysis of samples when the ICV is out-of-control must be taken with great care and in consultation with the QA department and the laboratory director. Any such action must be documented in an NCM.

10.3. Continuing calibration verification.

Note – A CCV standard need not be analyzed in tune periods when an initial calibration curve was analyzed.

10.3.1 To ensure calibration accuracy, during each analysis run a CCV/CCB. These should be run at a frequency of 10% or every two hours, whichever is more frequent.

11. PROCEDURE

11.1. Standard and Sample Preparation:

11.1.1. All calibration and calibration verification standards (ICV, ICB, CCV, CCB) are processed through the digestion procedure as well as the field samples.

11.1.2. Transfer 0, 0.2, 0.5, 1.0, 5.0 and 10.0 mL aliquots of the intermediate mercury standard (7.5) into a series of sample digestion bottles.

Note: Alternate volumes of standard may be prepared as long as the accuracy and final standard concentrations as detailed in Table I are maintained.

11.1.3. Prepare QC (ICV, ICB, CCV, CCB) using Intermediate Mercury QC Standard. For ICV and CCV concentrations, see Table 1.

11.1.4. Add reagent water to each standard and QC bottle to make a total volume of 10 mL. Continue preparation as described under 11.1.5 below.

11.1.5. Transfer 0.2 g of well mixed sample to a clean sample digestion bottle. Continue preparation as described under 11.1.5 below.

11.1.6. Water Bath protocol:

11.1.6.1. Add 5 mL of concentrated H_2SO_4 and 2.5 mL concentrated HNO_3.

11.1.6.2. Heat for 2 minutes in a water bath at 90-95 ° C.

11.1.6.3. Cool.

11.1.6.4. Add 50 mL of distilled water.

11.1.6.5. Add 15 mL of potassium permanganate solution.

11.1.6.6. Add 8 mL of potassium persulfate solution, mix thoroughly

11.1.6.7. Heat for 30 minutes in the water bath at 90-95 °C.

11.1.6.8. Cool.

11.1.6.9. Add 6 mL of sodium chloride-hydroxylamine sulfate solution to reduce the excess permanganate.

11.1.6.10. To each **standard** bottle: Add 50 mL of reagent water.
To each **sample** bottle: Add 60 mL of reagent water.

11.1.6.11. Continue as described under Section 11.2.

11.2. Sample Analysis:

11.2.1. Because of differences between various makes and models of CVAA instrumentation, no detailed operating instructions can be provided. Refer to the CVAA instrument manual for detailed setup and operation protocols.

11.2.2. Automated determination: Follow instructions provided by instrument manufacturer.

11.2.3. Perform a linear regression (for automated determination use a quadratic regression) analysis of the calibration standards by plotting maximum response of the standards vs. concentration of mercury. Determine the mercury concentration in the samples from the linear regression fit of the calibration curve. Calibration using computer or calculation based regression curve fitting techniques on concentration/response data is acceptable.

11.2.4. All measurements must fall within the defined calibration range to be valid. Dilute and reanalyze all samples for analytes that exceed the highest calibration standard.

11.2.5. If the sample results are negative and the absolute value of the negative result is greater than the reporting limit, the sample must be diluted and reanalyzed.

11.2.6. The samples must be allowed to cool to room temperature prior to analysis or a decrease in the response signal can occur.

11.2.7. Baseline correction is acceptable as long as it is performed after every sample or after the CCV and CCB; resloping is acceptable as long as it is immediately preceded and followed by a compliant CCV and CCB.

11.2.8. The following run sequence is consistent with 245.5 CLP-M, SOW ILMO3.0 requirements.

Instrument Calibration
ICV
ICB
CRA
CCV
CCB
10 samples
CCV
CCB
Repeat sequence of 10 samples between CCV/CCB pairs as required to complete run.
CCV
CCB

11.3. To facilitate the early identification of QC failures and samples requiring rerun it is strongly recommended that sample data be reviewed periodically throughout the run.

11.4. Guidelines are provided in the appendices on procedures to minimize contamination of samples and standards, preventive maintenance and troubleshooting.

11.5. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. For U.S. EPA contract analyses the Technical Project Officer must be contacted in advance for approval. The Nonconformance Memo shall be filed in the project file.

11.6. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

12. DATA ANALYSIS AND CALCULATIONS

12.1. ICV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(ICV)}{True(ICV)}\right)$$

12.2. CCV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(CCV)}{True(CCV)}\right)$$

12.3. Matrix spike recoveries are calculated according to the following equation:

$$\%\,R\; = \;100\left(\frac{SSR - SR}{SA}\right)$$

Where:

SSR = Spike Sample Result

SR = Sample Result

SA = Spike Added

12.4. The relative percent difference (RPD) of sample duplicates are calculated according to the following equation :

$$RPD = 100\left[\frac{|DU1 - DU2|}{\left(\frac{DU1 + DU2}{2}\right)}\right]$$

Where:

DU1 = Sample result

DU2 = Sample duplicate result

12.5. For automated determinations, the final concentration determined in solid samples when reported on a dry weight basis is calculated as follows:

$$mg/kg,\ dry\ weight = (C \times V \times D)/(W \times S)$$

Where:

C = Concentration (ug/L) from instrument readout

V = Volume of digestate (L)

D = Instrument dilution factor

W = Weight in g of wet sample digested
S = Percent solids/100

Note: A Percent Solids determination must be performed on a separate aliquot when dry weight concentrations are to be reported. If the results are to be reported on a wet weight basis, the "S" factor should be omitted from the above equation.

12.6. For manual (total) determinations, the final concentration determined in solid samples when reported on a dry weight basis is calculated as follows:

$$mg/kg,\ dry\ weight = (C)/(W \times S)$$

Where:
C = Concentration (ug) from instrument readout
W = Weight in g of wet sample digested
S = Percent solids/100

Note: A Percent Solids determination must be performed on a separate aliquot when dry weight concentrations are to be reported. If the results are to be reported on a wet weight basis, the "S" factor should be omitted from the above equation.

12.7. The LCS percent recovery is calculated according to the following equation:

$$\%R = 100\left(\frac{Found(LCS)}{True(LCS)}\right)$$

12.8. Appropriate factors must be applied to sample values if dilutions are performed.

12.9. Sample results should be reported with up to three significant figures in accordance with the following:

Result	Significant Figures
< IDL mg/kg	Report IDL u (2 sig figs)
> IDL and < 10.0 mg/kg	Report 2 sig figs
≥ 10.0 mg/kg	Report 3 sig figs

13. DATA ASSESSMENT AND ACCEPTANCE CRITERIA

The following represent data assessment for samples and acceptance criteria for QC measures. Corrective actions for any failure in QC measures are shown in Section 14.

13.1. QC sample acceptance criteria

13.1.1. Method Blank.

13.1.1.1. No target analytes may be present in the method blank above the reporting limit.

13.1.2. Laboratory Control Sample (LCS).

13.1.2.1. All control analytes must be within established control limits for accuracy (%Recovery) and precision (RPD). Exceptions are allowed only with QA and project management approval.

13.1.3. Matrix Spike/Matrix Spike Duplicate (MS/MSD).

13.1.3.1. All analytes should be within established control limits for accuracy (%Recovery) and precision (RPD). Deviations from this may be the results of matrix effects, which are confirmed by passing LCSs.

13.1.3.2. No specific corrective actions are required in the evaluation of the MS/MSD results provided that the batch LCS is in control. Analysts should use sound judgment in accepting MS/MSD results that are not within control limits, especially if the LCS results are borderline.

13.2. Sample result evaluation

13.2.1. Carryover. When a sample has a high response for a compound, there is a real possibility that some of the sample may carry over into the sample analyzed immediately afterward.

13.2.1.1. If a sample analyzed after a sample with high concentrations has negative results, carryover may have occurred and sample should be reanalyzed.

13.2.1.2. If a sample analyzed after a sample with high concentrations has positive results, or if the chromatographic profile resembles the previous sample, the results are questionable. This sample must be reanalyzed under conditions in which carryover can be confirmed to not have occurred.

13.2.2. Dilutions

13.2.2.1. If the response for any compound exceeds the working range of the analytical system, a dilution of the extract is prepared and analyzed. An appropriate dilution should be in the upper half of the calibration range.

13.2.2.2. If the initial diluted run has no hits or hits below 20% of the calibration range and the matrix allows for analysis at a lesser dilution, then the sample must be reanalyzed at a dilution targeted to bring the largest hit in the upper half of the calibration range.

14. CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

14.1. Method Blank.

14.1.1. The samples in the batch associated to the defective method blank are evaluated.

14.1.1.1. If the analyte found in the method blank is confirmed to not be present in any of the associated samples at any level, the contamination did not affect those samples. This represents a non-impact situation and no specific corrective action is taken. A nonconformance memo is written to notify project management of the situation for evaluation against project requirements.

14.1.1.2. If the analyte found in the method blank is confirmed to be present in one or more of the associated samples, the concentrations are compared.

14.1.1.2.1. If the concentration in the method blank exceeded 10% of concentration found in one or more samples, the prescribed corrective action is to re-analyze all affected samples.

14.1.1.2.2. If the concentration in the method blank was less than 10% of the concentration found in one or more samples, the sample can be reported by qualifying the affected analytes. A nonconformance memo is written to notify project management of the situation for evaluation against project requirements.

14.2. Laboratory control sample

14.2.1. If any control analyte is out of control for accuracy (% Recovery), the associated samples are evaluated.

14.2.1.1. If the recovery is biased high and the associated samples have no positive results for that analyte, a non-impact situation ensues. A nonconformance memo is written to notify project management of the situation for evaluation against project requirements.

15. CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

15.1. Method blanks

15.1.1. If there is insufficient sample to perform re-analysis, the analyst must notify the project manager for consultation with the client. In this situation, all associated samples are flagged with a "B" qualifier and appropriate comments in the narrative.

15.2. LCS

15.2.1. If the batch is not re-extracted and reanalyzed, the reasons for accepting the batch must be clearly presented in the project records and the report. Acceptable matrix spike recoveries are not sufficient justification to preclude re-extraction of the batch.

15.2.2. If re-extraction and reanalysis of the batch is not possible due to limited sample volume or other constraints, the LCS is reported, all associated samples are flagged, and appropriate comments are made in a narrative to provide further documentation.

15.3. Insufficient sample

15.3.1. If there is insufficient sample to repeat the analysis, the project manager is notified via NCM for consultation with the client.

16. METHOD PERFORMANCE

16.1. Each laboratory must have initial demonstration of performance data on file for each analyte of interest as described in Section 9.0.

16.2. Method performance is determined by the analysis of preparation blanks, laboratory control samples, matrix spikes and matrix duplicate samples. The matrix spike recovery should fall within +/- 25 % and the matrix spike duplicates should compare within 20% RPD. The preparation blank must meet the criteria specified in Section 9.2.4. The laboratory control sample should recover within the control limits established by the supplier.

16.3. Training Qualification:

The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

17. POLLUTION PREVENTION

This procedure will be carried out in a manner consistent with all applicable federal state and local regulations regarding pollution control and prevention. Specific controls due to the accidental release of hazardous materials can be found in the Chemical Hygiene Plan and facility attachments.

18. WASTE MANAGEMENT

Waste generated in this procedure will be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Coordinator should be contacted if additional information is required.

19. REFERENCES

19.1. USEPA Statement of Work for Inorganics Analysis, ILM03.0.

20. CLARIFICATIONS, MODIFICATIONS AND ADDITIONS TO REFERENCE METHOD

20.1. Modifications/Interpretations from reference method.

20.1.1. Modifications from 245.5 CLP-M

20.1.1.1. The SOW specifies the use of reagent water with a purity equivalent to ASTM Type II water. This SOP specifies the use of a Millipore DI system or equivalent to produce reagent water. This SOP requires that reagent water must be free of the analytes of interest as demonstrated through the analysis of preparation blanks.

20.1.1.2. Section 11.1.5.10 requires that 60 mL of reagent water be added to each sample bottle versus the 55 mL indicated in 245.5 CLP-M. This modification corrects for an error contained in the reference method which would otherwise result in an unequal dilution of the samples relative to the calibration standards.

20.2. Modifications from previous SOP: None

20.3. Facility Specific SOPs

Other than the routine operations facility specific SOPs, there are no other SOPs specific to this one.

20.4. Documentation and Record Management

The following documentation comprises a complete CVAA raw data package:

- Raw data (direct instrument printout)
- Run log printout from instrument software where this option is available or manually generated run log. (A bench sheet may be substituted for the run log as long as it contains an accurate representation of the analytical sequence and sufficient date and time information to verify that the QC time criteria were met).
- Data review checklist - See Appendix B
- Standards Documentation (source, lot, date).
- Copy of digestion log.
- Non-conformance summary (if applicable).

Figure 1. Solid Sample Preparation for Mercury - Water Bath Procedure

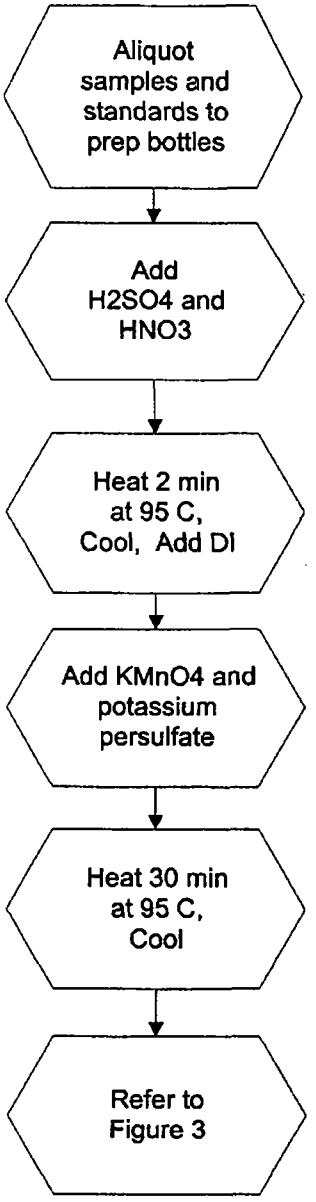
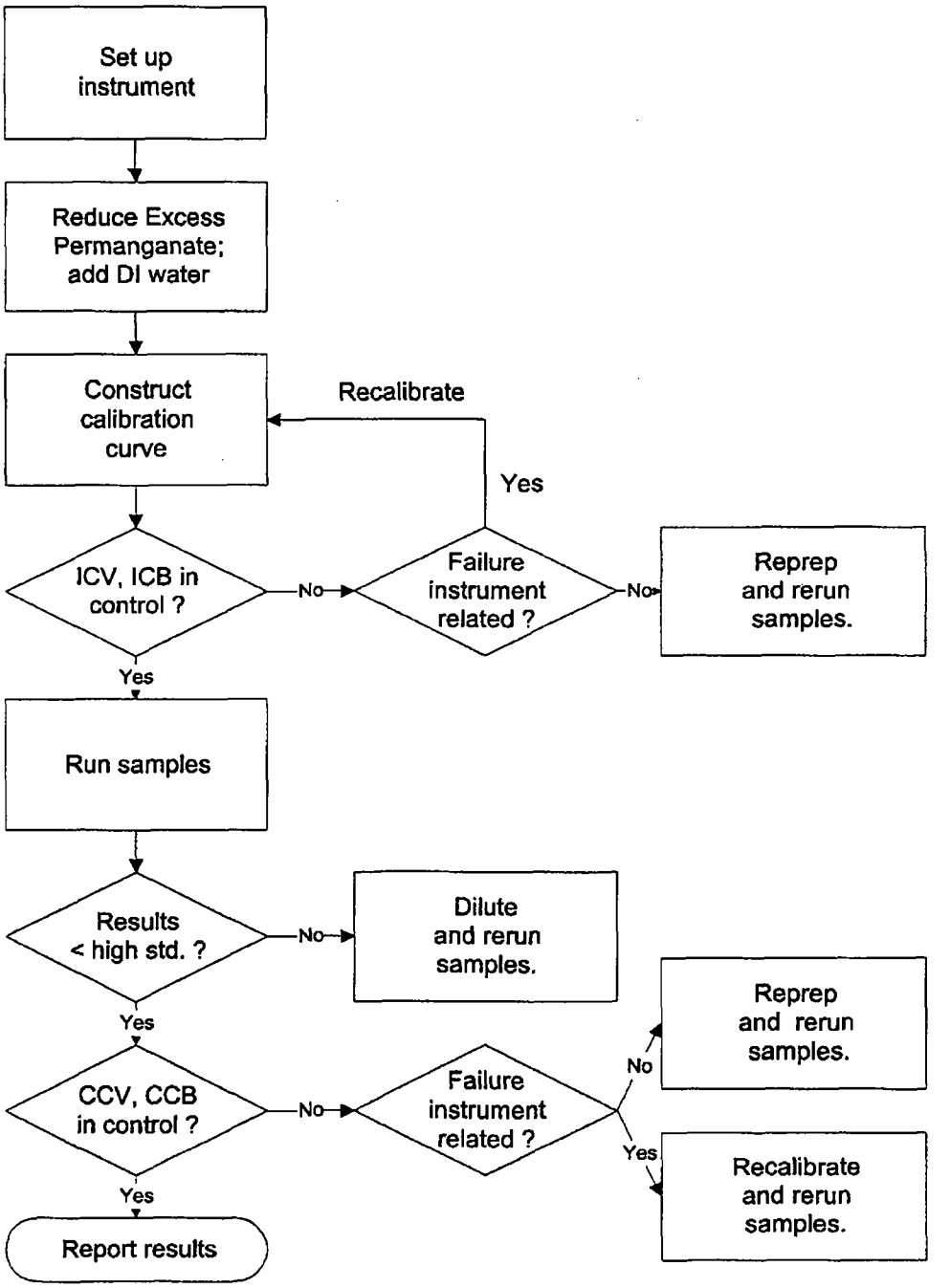


Figure 3. CVAA Mercury Analysis



APPENDIX A
TABLES

TABLE I. MERCURY REPORTING LIMITS, CALIBRATION STANDARD*, QC STANDARD AND SPIKING LEVELS

Soil RL (mg/kg)	0.1
Std 0 (ug/L)	0
Std 1 (ug/L)	0.2
Std 2 (ug/L)	0.5
Std 3 (ug/L)	1.0
Std 4 (ug/L)	5.0
Std 5 (ug/L) **	10
ICV (ug/L) ***	1.0 or 2.5 ***
CCV (ug/L)	2.5 or 5.0 ***
LCS (mg/kg) ***	Variable
MS (ug/L)	1.0

*** SOP specified calibration levels must be used unless prevented by the instrument configuration or client specific requirements. Deviations from specified calibration levels must be documented in the facility specific instrument operation SOP and must be approved by the facility technical manager and Quality Assurance Manager.**

**** Optional standard which may be used to extend the calibration range as allowed by the instrument configuration. If the instrument configuration prevents the use of 6 standards, the 2 ppb standard may be eliminated in favor of the 10 ppb standard.**

***** Concentration level dependent on high calibration standard used. CCV must be 50% of the high standard concentration and the ICV must be 20-25% of the high standard concentration.**

TABLE II. Summary Of Quality Control Requirements

QC PARAMETER	FREQUENCY *	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
ICV	Beginning of every analytical run.	80 - 120 % recovery.	Terminate analysis; Correct the problem; Recalibrate or reprep batch (see Section 9.2.8).
ICB	Beginning of every analytical run, immediately following the ICV.	The result must be within +/- CRDL from zero.	Terminate analysis; Correct the problem; Recalibrate or reprep batch (see Section 9.2.8).
CCV	Beginning and end of run and every 10 samples or every 2 hours, whichever is more frequent.	80 - 120 % recovery.	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCV or reprep batch (see Section 9.2.10).
CCB	Immediately following each CCV.	The result must be within +/- CRDL from zero.	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCB or reprep batch (see Section 9.2.10).
Preparation Blank	One per sample preparation batch of up to 20 samples.	The result must be less than or equal to the CRDL. Sample results greater than 10x the blank concentration are acceptable. Samples for which the contaminant is < RL do not require redigestion (See Section 9.2.4).	Redigest and reanalyze samples. Note exceptions under criteria section. See Section 9.2.4 for additional requirements.

*See Section 11.2.10 for exact run sequence to be followed.

TABLE II. Summary of Quality Control Requirements (Continued)

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
CRA	Beginning of each analytical run.	No criteria established by EPA at this time. In house criteria, +/- 50%.	Not Applicable.
Laboratory Control Sample (LCS)	One per sample preparation batch of up to 20 samples.	Soil LCS must be within limits established by supplier.	Terminate analysis; Correct the problem; Redigest and reanalyze all samples associated with the LCS (see Section 9.2.5).
Matrix Spike	One per SDG or each group of 20 samples of a similar matrix type.	75 - 125 % recovery .	Flag the data; no flag required if the sample level is > 4x the spike added. (see Section 9.2.6)
Matrix Duplicate	One per SDG or each group of 20 samples of a similar matrix type.	RPD ≤ 20% if results ≥ 5x CRDL or, ± CRDL if either result < 5x CRDL.	Flag the data. (see Section 9.2.7)

* See Section 11.2.10 for exact run sequence.

APPENDIX B
Hg DATA REVIEW CHECKLIST

Hg Data Review Checklist

Run/Project Information

Run Date: _____ **Analyst:** _____ **Instrument:** _____
Prep Batches Run: _____

Circle Methods used: 7470A / 245.1 : CORP-MT-0005 Rev 0 7471 / 245.5 : CORP-MT-0007 Rev 0
CLP - AQ : CORP-MT-0006 Rev 0 CLP - SOL : CORP-MT-0008 Rev 0

Review Items

A. Calibration/Instrument Run QC	Yes	No	N/A	2nd Level
1. Instrument calibrated per manufacturer's instructions and at SOP specified levels ?				
2. ICV/CCV analyzed at appropriate frequency and within control limits?				
3. ICB/CCB analyzed at appropriate frequency and within +/- RL or +/- CRDL (CLP)?				
4. CRA run (CLP only)?				
B. Sample Results				
1. Were samples with concentrations > the high calibration standard diluted and reanalyzed?				
2. All reported results bracketed by in control QC ?				
3. Sample analyses done within holding time?				
C. Preparation/Matrix QC				
1. LCS done per prep batch and within QC limits ?				
2. Method blank done per prep batch and < RL or CRDL (CLP) ?				
3. MS run at required frequency and within limits ?				
4. MSD or DU run at required frequency and RPD within SOP limits?				
D. Other				
1. Are all nonconformances documented appropriately ?				
2. Current IDL/MDL data on file?				
3. Calculations and Transcriptions checked for error ?				
4. All client/ project specific requirements met?				
5. Date of analysis verified as correct ?				

Analyst: _____ **Date:** _____
Comments:

_____

_____

2nd Level Reviewer : _____ **Date:** _____

_____

_____

APPENDIX C

TROUBLESHOOTING GUIDE

APPENDIX C. TROUBLESHOOTING GUIDE

Problem	Possible Cause
Poor or No Absorbance or Sensitivity Check failed	Incorrect wavelength Dirty windows Window loose Etched or dirty optics Bad lamp Not enough or no sample introduced Empty sample cup Incorrectly made standards Gas leak
Erratic Readings	Source lamp not aligned properly Lamp not prewarmed Contaminated reagents Contaminated glassware Drying tube saturated Bad lamp Leak in sample tubing Power fluctuations Air bubbles in tubing
Standards reading twice or half normal absorbance or concentration	Incorrect standard used Incorrect dilution performed Dirty cell

APPENDIX D
CONTAMINATION CONTROL GUIDELINES

APPENDIX D. CONTAMINATION CONTROL GUIDELINES

The following procedures are strongly recommended to prevent contamination:

All work areas used to prepare standards and spikes should be cleaned before and after each use.

All glassware should be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water.

Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.

Powdered or Latex Gloves must not be used in the metals laboratory since the powder contains silica and zinc, as well as other metallic analytes. Only vinyl or nitrile gloves should be used in the metals laboratory.

Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.

Autosampler trays should be covered to reduce the possibility of contamination. Trace levels of elements being analyzed in the samples can be easily contaminated by dust particles in the laboratory.

The following are helpful hints in the identification of the source of contaminants:

Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.

Improper cleaning of glassware can cause contamination.

Separate glassware if an unusually high sample is analyzed and soak with nitric acid prior to routine cleaning.

APPENDIX E
PREVENTIVE MAINTENANCE

APPENDIX E. PREVENTIVE MAINTENANCE

A maintenance log is used to record when maintenance is performed on instruments. When an instrument problem occurs indicate the date, time and instrument number, then identify the problem and corrective action in the maintenance log.

The following procedures are required to ensure that that the instrument is fully operational.

Cold Vapor Atomic Absorption (Leeman PS 200) [1]

Daily	Annually
Check Hg lamp intensity.	Change Hg lamp.
Check aperture.	Clean cell
Check argon flow.	
Check tubing.	
Check drain.	
Check liquid/gas separator	

Cold Vapor Atomic Absorption (PE 5000) [1]

Daily	Monthly
Clean aspirator by flushing with DI water.	Clean cell in aqua regia.
Check tubing and replace if needed.	Clean aspirator in aqua regia.
Clean windows with methanol.	
Change silica gel in drying tube.	
Check argon gas supply.	
Adjust lamp.	

Attachment QAPP-C4

GC/MS Analysis
Methods 8270 and 625

SOP No.:	**CORP-MS-0001 STL**
Revision No.:	2.3
Revision Date:	10/01/01
Page:	1 of 47
Implementation Date:	10/15/01

Controlled Copy Number: _____

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STL ST. LOUIS STANDARD OPERATING PROCEDURE

TITLE: GC/MS ANALYSIS BASED ON METHODS 8270C AND 625

(SUPERSEDES: REVISION 2.2)

Prepared by: _____

Approved by: _____
Technical Specialist

Approved by: _____
Quality Assurance Manager

Approved by: _____
Environmental Health and Safety Coordinator

Approved by: _____
Laboratory Director

1. SCOPE AND APPLICATION

1.1. This SOP is applicable to the determination of the concentration of semivolatile organic compounds in extracts prepared from solid and aqueous matrices

1.2. This SOP is based on SW-846 Method 8270C. The modifications presented in Attachment A may be followed for analysis of wastewater following method 625. Direct injection of a sample may be used in limited applications. Refer to Tables 1, 2, 3 and 4 for the list of compounds applicable for this method. Note that the compounds are listed in approximate retention time order. Additional compounds may be amenable to this method. If non-standard analytes are required, they must be validated by the procedures described in Section 16 before sample analysis.

1.3. The following compounds may require special treatment when being determined by this method:

- Benzidine can be subject to oxidative losses during solvent concentration and exhibits poor chromatography. Neutral extraction should be performed if this compound is expected.
- Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reaction in acetone solution, and photochemical decomposition.
- N-Nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be distinguished from diphenylamine.
- Pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, 4,6-dinitro-2-methylphenol, 4-chloro-3-methylphenol, benzoic acid, 2-nitroaniline, 3-nitroaniline, 4-chloroaniline, and benzyl alcohol are subject to erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material.
- Hexachlorophene may not be amenable to analysis by this method.
- 3-Methylphenol cannot be separated from 4-methylphenol by the conditions specified in this method.

1.4. The standard reporting limit (SRL) of this method for determining an individual compound is approximately 0.33 mg/kg (wet weight) for soil/sediment samples, 1 - 200 mg/kg for wastes (dependent on matrix and method of preparation), and 10 µg/L for groundwater samples. Some compounds have higher reporting limits. Refer to Tables 1 and 2 for specific SRLs. Reporting limits will be proportionately higher for sample extracts that require dilution.

1.5. Method detection limits are maintained in the Information Management System (QuantIMS). Because of their dynamic nature, they are not specifically listed in this document, but can be retrieved at any time using TraQAr tools.

1.6. Quality control limits (accuracy and precision for spikes) are also maintained in QuantIMS, and are also dynamic. Therefore, they are not specifically listed in this document, but can be retrieved at any time using TraQAr tools.

1.7. Additional compounds may be amenable to this method. The minimum requirement for non-standard analytes is that the reporting limit be set at the lowest required concentration that can actually be detected by the instrument, and when an MDL study can not be conducted, the MDL be set equal to the reporting limit.

2. SUMMARY OF METHOD

2.1. Aqueous samples are extracted with methylene chloride using a separatory funnel, a continuous extractor or Accelerated One-Step™. Solid samples are extracted with methylene chloride / acetone using sonication, soxhlet, accelerated soxhlet or pressurized fluid extraction. Waste dilution is used for samples that are miscible with the solvent. The extract is dried, concentrated to a volume of 1 ml, and analyzed by GC/MS.

Extraction procedures are detailed in SOP# CORP-OP-0001. Qualitative identification of the parameters in the extract is performed using the retention time and the relative abundance of characteristic ions. Quantitative analysis is performed using the internal standard technique with a single characteristic ion.

3. DEFINITIONS

3.1. See policy QA-003 for definitions

3.2. CCC (Calibration Check Compounds) - A subset of target compounds used to evaluate the calibration stability of the GC/MS system. A maximum percent deviation of the CCC's is specified for calibration acceptance.

3.3. SPCC (System Performance Check Compounds) - Target compounds designated to monitor chromatographic performance, sensitivity, and compound instability or degradation on active sites. Minimum response factors are specified for acceptable performance.

3.4. Batch - The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. The Quality Control batch must contain a matrix spike / spike duplicate (MS/MSD), a Laboratory Control Sample (LCS), and a method blank. In some cases, at client request, the MS/MSD may be replaced with a matrix spike and sample duplicate or with duplicate LCS if not enough sample is supplied for MS/MSD. Batches are defined at the sample preparation stage. Batches should be kept together through the whole analytical process to the extent possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. Refer to the QC Program document (QA-003) for further details of the batch definition.

3.5. Method Blank - An analytical control consisting of all reagents, internal standards and surrogate standards, that is carried through the entire analytical procedure. The method blank is used to define the level of laboratory background and reagent contamination.

3.6. LCS (Laboratory Control Sample) - A blank spiked with the parameters of interest that is carried through the entire analytical procedure. Analysis of this sample with acceptable recoveries of the spiked materials demonstrates that the laboratory techniques for this method are acceptable.

3.7. MS (Matrix Spike)- aliquot of a matrix (water or soil) fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.

3.8. MSD (Matrix Spike Duplicate)- a second aliquot of the same sample as the matrix spike (above) that is spiked in order to determine the precision of the method.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. If an interference is detected it is necessary to determine if the source of interference is in the preparation and/or cleanup of the samples; then take corrective action to eliminate the problem.

4.2. The use of high purity reagents, solvents, and gases helps to minimize interference problems.

4.3. Matrix interferences may be caused by contaminants that are coextracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature of the sample.

4.4. Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed with solvent between samples. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of solvent to check for cross contamination.

4.5. Phthalate contamination is commonly observed in this analysis and its occurrence should be carefully evaluated as an indicator of a contamination problem in the sample preparation step of the analysis.

5. SAFETY

5.1. Procedures shall be carried out in a manner that protects the health and safety of all associates.

5.2. The Chemical Hygiene Plan (CHP) gives details about the specific health and safety practices which are to be followed in the laboratory area. Personnel must receive training in the CHP, including the written Hazard Communication plan, prior to working in the laboratory. Consult the CHP, the Health and Safety Policies and Procedures Manual, and available Material Safety Data Sheets (MSDS) prior to using the chemicals in the method.

5.3. Consult the Health and Safety Policies and Procedures Manual for information on Personal Protective Equipment. Eye protection that satisfies ANSI Z87.1 (as per the Chemical Hygiene Plan), laboratory coat and appropriate gloves must be worn while samples, standards, solvents and reagents are being handled. Disposable gloves that have become contaminated will be removed and discarded; other gloves will be cleaned immediately. VITON gloves may be worn when halogenated solvents are used for extractions or sample preparation. Nitrile gloves may be used when other solvents are handled.

5.4. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the MSDS files maintained in the laboratory. The following specific hazards are known:

5.4.1. Chemicals that have been classified as **carcinogens**, or potential carcinogens, under OSHA include: Benzo(a)anthracene, benzidine, 3,3'-dichlorobenzidine, benzo(a)pyrene, dibenzo(a,h)anthracene, and n-nitrosodimethylamine. Primary standards should be purchased in solution. If neat materials must be obtained, they shall be handled in a hood.

5.5. Exposure to chemicals will be maintained **as low as reasonably achievable,** therefore, unless they are known to be non-hazardous, all samples will be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.6. The preparation of all standards and reagents and glassware cleaning procedures that involve solvents will be conducted in a fume hood with the sash closed as far as the operations will permit or by other means of mechanical ventilation.

5.7. All work must be stopped in the event of a known, or potential compromise to the health or safety of any associate. The situation must be reported **immediately** to a laboratory supervisor.

5.8. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices outlined in the Health and Safety Manual. These employees must have training on the hazardous waste disposal practices initially upon assignment of these tasks, followed by an annual refresher training.

6. EQUIPMENT AND SUPPLIES

6.1. Gas Chromatograph/Mass Spectrometer System: An analytical system complete with a temperature-programmable gas chromatograph suitable for split/splitless injection and all required accessories, including syringes, analytical columns, and gases. The capillary column should be directly coupled to the source.

6.2. Column: 30 m x 0.32 mm I.D. (or 0.25 mm I.D.) 0.5-µm film thickness silicon-coated fused-silica capillary column (J & W Scientific DB-5.625 or equivalent). Alternate columns are acceptable if they provide acceptable performance.

6.3. Mass Spectrometer: Capable of scanning from 35 to 500 AMU every one second or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be capable of

producing a mass spectrum for decafluorotriphenylphosphine (DFTPP) which meets all of the criteria in Table 6 when 50 ng of the GC/MS tuning standard is injected through the GC.

6.4. GC/MS Interface: Any GC-to-MS interface that gives acceptable calibration points and achieves acceptable tuning performance criteria may be used.

6.5. Data System: A computer system must be interfaced to the mass spectrometer. The system must allow the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that can search any GC/MS data file for ions of a specific mass and that can plot such ion abundances versus time or scan number. This type of plot is defined as the Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between specified time or scan-number limits. The most recent version of the EPA/NIH Mass Spectral Library is recommended.

6.6. Syringe: 10 µL Hamilton Laboratory grade syringes or equivalent.

6.7. Carrier gas: Ultra high purity helium.

7. REAGENTS AND STANDARDS

7.1. Reagents

7.1.1. All reagent preparation is documented in the reagent logbook. All reagents are labeled with their unique ID, name (and concentration, if applicable) of the reagent, the date prepared and the expiration date.

7.2. Standards

7.2.1. All standards preparation, documentation and labeling must follow the requirements of STL-QA-0002.

7.2.2. At a minimum, a five point calibration curve is prepared. The low point should be at or below the reporting limit. Refer to Tables 12 and 13 for typical calibration levels for all analytes. Other calibration levels may be used, depending on instrument capability, but the low standard must support the reporting limit and the high standard defines the range of the calibration.

7.2.3. An Internal Standard (IS) solution is prepared. Compounds in the I.S. Mix are: acenaphthene-d10, chrysene-d12, 1,4-dichlorobenzene-d4, naphthalene-d8, perylene-d12, and phenanthrene-d10.

7.2.4. Internal Standards are added to all standards and extracts to result in 40ng injected onto the column. For example, if the volume of an extract used was 200 µL, 10 µL of a 400 µg/ml internal standard solution would be added for a 2 µL injection.

7.2.5. Surrogate Standard Spiking Solution: Prepare as indicated in the preparative methods. See appropriate preparation SOP. Surrogate compounds and levels are listed in Table 11.

7.2.6. GC/MS Tuning Standard: A methylene chloride solution containing 50 µg/ml of decafluorotriphenylphosphine (DFTPP) is prepared.

7.2.7. Laboratory Control Spiking Solution: Prepare as indicated in the preparative methods. See appropriate preparation SOP. LCS compounds and levels are listed in Tables 9 and 10.

7.2.8. Matrix Spike Solution: Prepare as indicated in the preparative methods. See preparation SOP. The matrix spike compounds and levels are the same as the LCS compounds.

7.2.9. The standards listed in 7.2.2 to 7.2.8 should be refrigerated at $\leq 6°C$ when not in use. Refrigeration at -10°C to -20°C may be used if it can be demonstrated that analytes do not fall out of solution at this temperature. The standards must be replaced at least once a year. The continuing calibration standard must be replaced every week and is stored at $\leq 6°C$.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Samples are stored at 4 ± 2°C. Samples and extracts should be stored in suitable glass containers with Teflon lined caps. (Extracts will normally be stored for 30 days after invoicing.)

8.2. Water samples are extracted within seven days of sampling and the extracts are analyzed within forty days of extraction. Solids, sludges, and organic liquids are extracted within fourteen days of sampling and the extracts are analyzed within forty days of extraction.

9. QUALITY CONTROL

9.1. QC requirements

9.1.1. Each analytical batch may contain up to 20 environmental samples, a method blank, and a single Laboratory Control Sample (LCS) and an MS/MSD pair. In the event that there is not sufficient sample to analyze an MS/MSD, an LCS duplicate (LCSD) is prepared and analyzed.

9.1.1.1. Samples that have assigned QC limits different than the standard limits contained in QuantIMS QC code 01 must be batched separately, but can share the same QC samples.

9.1.1.2. Additional MS/MSDs do not count towards the 20 samples in an analytical batch.

9.1.1.3. A method blank must be included with each batch of samples

9.1.1.4. The LCS is spiked with all of the standard target compounds and is used to monitor the accuracy of the analytical process, independent of matrix effects

9.1.1.5. All LCS, MS/MSD and surrogate results -- whether they pass criteria or not -- are uploaded into 's QuantIMS system for maintenance and periodic update of limits.

9.1.2. Instrument conditions must be the same for all standards, samples and QC samples.

9.1.3. All data will be reviewed by the analyst (1st review level) and then by a peer or supervisor (2nd level review).

9.2. Documentation

9.2.1. Initial Demonstration of Capability

9.2.1.1. For the standard analyte list, the initial demonstration and method detection limit (MDL) studies described in section 16 must be acceptable before analysis of samples may begin. Refer to the flow chart in Section 20.4.1.

9.2.1.2. For non-standard analytes an IDOC and MDL study must be performed and calibration curve generated before analyzing any samples, unless lesser requirements are previously agreed to with the client. In any event, the minimum initial demonstration required is analysis of an extracted standard at the reporting limit and a single point calibration.

9.2.2. Control Limits

In-house historical control limits must be determined for surrogates, matrix spikes, and laboratory control samples (LCS). These limits must be determined at least annually. The recovery limits are mean recovery +/- 3 standard deviations for surrogates, MS and LCS Precision limits for matrix spikes / matrix spike duplicates are mean relative percent difference +/- 3 standard deviations.

9.2.2.1. These limits do not apply to dilutions (except for tests without a separate extraction), but surrogate and matrix spike recoveries will be reported unless the dilution is more than 5X.

9.2.2.2. All surrogate, LCS, and MS recoveries (except for dilutions) must be entered into QuantIMS (when available) or other database so that accurate historical control limits can be generated. For tests without a separate extraction, surrogates and matrix spikes will be reported for all dilutions.

9.2.2.3. Refer to the QC program document (QA-003) for further details of control limits.

9.2.3. Method Blank

A method blank is prepared and analyzed with each batch of samples. The method blank consists of reagent water for aqueous samples, and sodium sulfate for soil samples (Refer to SOP No. CORP-OP-0001 for details). Surrogates are added and the method blank is carried through the entire analytical procedure. The method blank must not contain any analyte of interest at or above the reporting limit (including common laboratory contaminants).

- When concentrations of any analyte of interest are at or above the reporting limit, the laboratory must take corrective action to locate and reduce the source of contamination.
- Reanalysis of any samples with reportable concentrations of analytes found in the method blank is required unless other actions are agreed with the client.
- If there is no target analyte greater than the RL in the samples associated with an unacceptable method blank or if the concentration of the analyte found in the blank is found in a sample at greater than 20 times the concentration in the blank, the data may be reported with qualifiers. Such action should be taken in consultation with the client.

9.2.3.1. The method blank must have acceptable surrogate recoveries. If surrogate recoveries are not acceptable, the data must be evaluated to determine if the method blank has served the purpose of demonstrating that the analysis is free of contamination. If surrogate recoveries are low and there are reportable analytes in the associated samples, re-extraction of the blank and affected samples will normally be required. Consultation with the client should take place.

9.2.3.2. If reanalysis of the batch is not possible due to limited sample volume or other constraints, the method blank is reported, all associated samples are flagged with a "B", and appropriate comments may be made in a narrative to provide further documentation.

9.2.3.3. Refer to the QC Program document (QA-003) for further details of the corrective actions.

9.2.3.4. Sample results are NOT blank subtracted unless specific requests and arrangements have been made with a client or agency.

9.2.4. Instrument Blank

9.2.4.1. Instruments must be evaluated for contamination during each 12 hour analytical run. This may be accomplished by analysis of a method blank. If a method blank is not available, an instrument blank must be analyzed. An instrument blank consists of methylene chloride with the internal standards added. It is evaluated in the same way as the method blank.

9.2.5. Laboratory Control Sample (LCS)

9.2.5.1. A laboratory control sample (LCS) is prepared and analyzed with every batch of samples. All analytes must be within established control limits. The LCS is spiked with the compounds listed in Tables 9 and 10 unless specified by a client or agency. The compounds must be spiked at a concentration equivalent to 100 or 150 ng on-column depending on the analyte.

9.2.5.2. If any analyte in the LCS is outside the laboratory established historical control limits, corrective action must occur. Corrective action may include re-extraction and reanalysis of the batch.

- If the batch is not re-extracted and reanalyzed, the reasons for accepting the batch must be clearly presented in the project records and the report. (An example of acceptable reasons for not reanalyzing might be that the matrix spike and matrix spike duplicate are acceptable, and sample surrogate recoveries are good, demonstrating that the problem was confined to the LCS).
- If re-extraction and reanalysis of the batch is not possible due to limited sample volume or other constraints, the LCS is reported, all associated samples are flagged, and appropriate comments are made in a narrative to provide further documentation.

9.2.5.3. Ongoing monitoring of the LCS provides evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision.

9.2.5.4. Real-time control limits may be used for sample surrogate evaluations and/or MS/MSD spike evaluations. Refer to SOP STL-QA-0014.

9.2.6. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

A matrix spike/matrix spike duplicate (MS/MSD) is prepared and analyzed with every batch of samples. The MS/MSD is spiked with the same subset of analytes as the LCS (See Tables 9 and 10). Compare the percent recovery and relative percent difference (RPD) to that in the laboratory specific historically generated limits.

- If any individual recovery or RPD falls outside the acceptable range, corrective action must occur. The initial corrective action will be to check the recovery of that analyte in the Laboratory Control Sample (LCS). Generally, if the recovery of the analyte in the LCS is within limits, then the laboratory operation is in control and analysis may proceed. The reasons for accepting the batch must be documented.
- If the recovery for any component is outside QC limits for both the Matrix spike / spike duplicate and the LCS, the laboratory is out of control and corrective action must be taken. Corrective action will normally include repreparation and reanalysis of the batch.
- If a MS/MSD is not possible due to limited sample, then a LCS duplicate should be analyzed. RPD of the LCS and LCSD are compared to the matrix spike limits.
- The matrix spike / duplicate must be analyzed at the same dilution as the unspiked sample, even if the matrix spike compounds will be diluted out.

9.2.7. Surrogates

9.2.7.1. Every sample, blank, and QC sample is spiked with surrogate standards. Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits. Surrogate compounds must be spiked at either 100 or 150 ng on-column, depending on the surrogate. The compounds routinely included in the surrogate spiking solution, along with recommended standard concentrations, are listed in Table 11.

9.2.7.2. If any surrogates are outside limits the following corrective actions must take place (except for dilutions):

- Check all calculations for error.
- Ensure that instrument performance is acceptable.

- Recalculate the data and/or reanalyze the extract if either of the above checks reveal a problem.
- Re-extract and reanalyze the sample or flag the data as "Estimated Concentration" if neither of the above resolves the problem.
  The decision to reanalyze or flag the data should be made in consultation with the client. It is only necessary to reprepare / reanalyze a sample once to demonstrate that poor surrogate recovery is due to matrix effect, unless the analyst believes that the repeated out of control results are not due to matrix effect.

9.2.7.3. If the sample with surrogate recoveries outside the recovery limits was a sample used for an MS/MSD and the surrogate recoveries in the MS/MSD are also outside of the control limits, then the sample, the MS, and the MSD do not require reanalysis as this phenomenon would indicate a possible matrix problem.

9.2.7.4. If the sample is reanalyzed and the surrogate recoveries in the reanalysis are acceptable, then the problem was within the analyst's control and only the reanalyzed data should be reported. (Unless the reanalysis was outside holding times, in which case reporting both sets of results may be appropriate.)

9.2.7.5. If the reanalysis does confirm the original results, the original analysis is reported and the data flagged as estimated due to matrix effect.

9.2.8. Quality Assurance Summaries

9.2.8.1. Certain clients may require specific project or program QC which may supercede these method requirements. These requirements should be addressed in the Client Requirement Checklist.

9.3. Procedural Variations

9.3.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of the analyst to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

9.3.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

9.4. Nonconformance and Corrective Action

Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the facility QA Manager.

9.5. QC Program

Further details of QC and corrective action guidelines are presented in the QC Program policy document (QA-003).

10. CALIBRATION AND STANDARDIZATION

10.1. Summary

10.1.1. The instrument is tuned for DFTPP, calibrated initially with a five-point calibration curve, and verified each 12-hour shift with one or more continuing calibration standard(s). Recommended instrument conditions are listed in Table 5.

10.2. All standards and extracts are allowed to warm to room temperature before injecting.

10.3. Instrument Tuning

At the beginning of every twelve hour shift when analyses are to be performed, the GC/MS system must be checked to see if acceptable performance criteria (Table 6) is achieved for DFTPP (decafluorotriphenylphosphine).

10.3.1. Inject 50 ng of the GC/MS tuning standard (Section 7.2.6) into the GC/MS system. Obtain a background-corrected mass spectra of DFTPP and confirm that all the key m/z criteria in Table 6 are achieved. If all the criteria are not achieved, the analyst must retune the mass spectrometer and repeat the test until all criteria are achieved. The performance criteria must be achieved before any samples, blanks, or standards are analyzed.

10.4. Calibration

10.4.1. Initial and continuing calibrations will be based on a linear response factor model in all cases except those that are pre-approved by the house QC officer for quadratic calibration. In the case of linear calibrations, response factors will be used to determine percent difference where required. In the case of quadratic calibrations, correlation factors will be used for calibration curve evaluation.

10.5. Initial Calibration

10.5.1. Internal Standard Calibration Procedure: Internal standards are listed in Table 7. Use the base peak m/z as the primary m/z for quantitation of the standards. If interferences are noted, use one of the next two most intense masses for quantitation.

10.5.2. Compounds should be assigned to the IS with the closest retention time.

10.5.3. Prepare calibration standards at a minimum of five concentration levels for each parameter of interest. Six standards must be used for a quadratic least squares calibration. It may also be useful to analyze six calibration levels and use the lower five for most analytes and the upper five for analytes that have poor response. Add the internal standard mixture to result in 40 ng on column. (For example, if the volume of the calibration standard used is 1 ml, add 50 µL of the 400 µg/ml internal standard solution for a 2 µL injection). The concentrations of all analytes are listed in tables 12 and 13.

10.5.4. Analyze each calibration standard and tabulate the area of the primary characteristic m/z against concentration for each compound and internal standard. Calculate response factors (RF), average response factors, and the percent RSD of the response factors for each compound using the equations in section 12 and verify that the CCC and SPCC criteria in section 10.4.5 and 10.4.6 are met. **No sample analysis may be performed unless these criteria are met.**

10.5.5. System Performance Check Compounds (SPCCs): The minimum average RF for semivolatile SPCCs is 0.050. If the minimum response factors are not met, the system must be evaluated and corrective action must be taken before sample analysis begins. Some possible problems are standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system. This check must be met before analysis begins.

SPCC Compounds:

N-nitroso-di-n-propylamine
Hexachlorocyclopentadiene
2,4-Dinitrophenol
4-Nitrophenol

10.5.6. Calibration Check Compounds (CCCs): The %RSD of the response factors for each CCC in the initial calibration must be less than 30% for the initial calibration to be considered valid. This criterion must be met before sample analysis begins. Problems similar to those listed under SPCCs could affect this criterion.

10.5.6.1. If none of the CCCs are required analytes, project specific calibration specifications must be agreed with the client.

10.5.6.2. CCC Compounds:

Phenol
Acenaphthene
1,4-Dichlorobenzene
N-nitrosodiphenylamine
2-Nitrophenol
Pentachlorophenol
2,4-Dichlorophenol
Fluoranthene
Hexachlorobutadiene
Di-n-octylphthalate
4-Chloro-3-methylphenol
Benzo(a)pyrene
2,4,6-Trichlorophenol

10.5.7. If the average of all %RSDs in the initial calibration is $\leq$ 15%, then all analytes may use average response factor for calibration.

10.5.7.1. If the software in use is capable of routinely reporting curve coefficients for data validation purposes, and the necessary calibration reports can be generated, then the analyst should evaluate analytes with %RSD > 15% for calibration on a curve. If it appears that substantially better accuracy would be obtained using quantitation from a curve then the appropriate curve should be used for quantitation.

10.5.7.2. If the average of all the %RSDs in the initial calibration is > 15%, then calibration on a curve must be used for those analytes with %RSD > 15%. Linear or quadratic curve fits may be used. Use of $1/Concentration^2$ weighting is recommended to improve the accuracy of quantitation at the low end of the curve. The analyst should consider instrument maintenance to improve the linearity of response. If Relative Standard Error (RSE) is used to evaluate the curve it must be better than 15%. Otherwise the correlation coefficient (coefficient of determination for non-linear curves) must be $\geq$ 0.990.

10.5.8. Weighting of data points

In a linear or quadratic calibration fit, the points at the lower end of the calibration curve have less weight in determining the curve generated than points at the high concentration end of the curve. However, in environmental analysis, accuracy at the low end of the curve is very important. For this reason it is preferable to increase the weighting of the lower concentration points. $1/Concentration^2$ weighting (often called $1/X^2$ weighting) will improve accuracy at the low end of the curve and should be used if the data system has this capability.

10.5.9. If time remains in the 12 hour period initiated by the DFTPP injection before the initial calibration, samples may be analyzed. Otherwise, proceed to continuing calibration.

10.5.10. Quantitation is performed using the calibration curve or average response factor from the initial curve, not the continuing calibration.

10.6. Continuing Calibration

10.6.1. At the start of each 12-hour period, the GC/MS tuning standard must be analyzed. A 50 ng injection of DFTPP must result in a mass spectrum for DFTPP which meets the criteria given in Table 6.

10.6.2. Following a successful DFTPP analysis the continuing calibration standard(s) are analyzed. The standards must contain all semivolatile analytes, including all required surrogates. A mid level calibration standard is used for the continuing calibration.

10.6.3. The following criteria must be met for the continuing calibration to be acceptable:

- The SPCC compounds must have a response factor of ≥ 0.05.
- The percent difference of the CCC compounds from the initial calibration must be $\leq$ 20%. (see section 12 for calculations) In addition, the percent difference or drift of all analytes must be $\leq 50\%$, with allowance being made for up to six target compounds to have percent drift greater than 50%.
- The internal standard response must be within 50-200% of the response in the mid level of the initial calibration.
- The internal standard retention times must be within 30 seconds of the retention times in the mid-level of the initial calibration.

10.6.3.1. If none of the CCCs are required analytes, project specific calibration specifications must be agreed with the client.

10.6.4. Once the above criteria have been met, sample analysis may begin. Initial calibration average RFs (or the calibration curve) will be used for sample quantitation, not the continuing calibration RFs. Analysis may proceed until 12 hours from the injection of the DFTPP have passed. (A sample *injected* less than 12 hours after the DFTPP is acceptable.)

11. PROCEDURE

11.1. Sample Preparation

Samples are prepared following SOP CORP-OP-0001.

11.2. Sample Analysis Procedure

11.2.1. Calibrate the instrument as described in section 10. Depending on the target compounds required by the client, it may be necessary to use more than one calibration standard.

11.2.2. All samples must be analyzed using the same instrument conditions as the preceeding continuing calibration standard.

11.2.3. Add internal standard to the extract to result in 40 ng injected on column (for example, 25 µL internal standard solution in 0.5 mL of extract for a 2 µL injection). Mix thoroughly before injection into the instrument.

11.2.4. Inject the sample extract into the GC/MS system using the same injection technique as used for the standards.

11.2.5. The data system will determine the concentration of each analyte in the extract using calculations equivalent to those in section 12. Quantitation is based on the initial calibration, not the continuing calibration.

11.2.6. Identified compounds are reviewed for proper integration. Manual integrations are performed if necessary and are documented by the analyst or automatically by the data system.

11.2.7. Target compounds identified by the data system are evaluated using the criteria listed in Section 12.1.

11.2.8. Library searches of peaks present in the chromatogram that are not target compounds (Tentatively Identified Compounds, TIC) may be performed if required by the client. They are evaluated using the criteria in section 12.3.

11.3. Dilutions

If the response for any compound exceeds the working range of the GC/MS system, a dilution of the extract is prepared and analyzed. An appropriate dilution should be in the upper half of the calibration range. Samples may be screened to determine the appropriate dilution for the initial run. If the initial diluted run has no hits or hits below 20% of the calibration range and the matrix allows for analysis at a lesser dilution, the sample must be reanalyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

11.3.1. Guidance for Dilutions Due to Matrix

If the sample is initially run at a dilution and the baseline rise is less than the height of the internal standards, or if individual non-target peaks are less than two times the height of the internal standards, the sample should be reanalyzed at a more concentrated dilution. This requirement is approximate and subject to analyst judgment. For example, samples containing organic acids may need to be analyzed at a higher dilution to avoid destroying the column.

11.3.2. Reporting Dilutions

The most concentrated dilution with no target compounds above the calibration range will be reported. Other dilutions will only be reported at client request.

11.4. Perform all qualitative and quantitative measurements. When the extracts are not being used for analyses, refrigerate them at 4 ± 2°C, protected from light in screw cap vials equipped with unpierced Teflon lined septa.

11.5. Retention time criteria for samples

If the retention time for any internal standard changes by more than 0.5 minutes from the last continuing calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Reanalysis of samples analyzed while the system was malfunctioning is required.

11.5.1. If the retention time of any internal standard in any sample varies by more than 0.1 minute from the preceding continuing calibration standard, the data must be carefully evaluated to ensure that no analytes have shifted outside their retention time windows.

11.6. Percent Moisture

Analytical results may be reported as dry or wet weight, as required by the client. Percent moisture must be determined if results will be reported as dry weight. Refer to the facility specific SOP for determination of percent moisture.

11.7. Troubleshooting Guide

11.7.1. Daily Instrument Maintenance

In addition to the checks listed in the instrument maintenance schedule in the QAMP, the following daily maintenance should be performed.

- Clip Column as necessary.
- Install new or cleaned injection port liner as necessary.
- Install new septum as necessary.
- Perform mass calibration as necessary.

11.7.2. Major Maintenance

11.7.2.1. A new initial calibration is necessary following major maintenance. Major maintenance includes changing the column, cleaning the ion volume or repeller, cleaning the source, and replacing the multiplier. Refer to the manufacturer's manual for specific guidance.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Qualitative identification

An analyte is identified by retention time and by comparison of the sample mass spectrum with the mass spectrum of a standard of the suspected compound (standard reference spectrum). Mass spectra for standard reference may be obtained on the user's GC/MS by analysis of the calibration standards or from the NBS library. Two criteria must be satisfied to verify identification: (1) elution of sample component at the same GC retention time as the standard component; and (2) correspondence of the sample component and the standard component characteristic ions. (Note: Care must be taken to ensure that spectral distortion due to co-elution is evaluated.)

- The sample component retention time must compare to within ± 0.2 min. of the retention time of the standard component. For reference, the standard must be run within the same twelve hours as the sample.
- All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) should be present in the sample spectrum.
- The characteristic ions of a compound must maximize in the same scan or within one scan of each other.
- The relative intensities of ions should agree to within ±30% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 20% and 80%.)

12.1.1. If a compound cannot be verified by all the above criteria, but in the technical judgment of the analyst the identification is correct, the analyst shall report that identification and proceed with quantitation.

12.2. Mass chromatogram searches.

Certain compounds are unstable in the calibration standard and cannot be calibrated in the normal way. In particular, the compound hexachlorophene (CAS 70-30-4) falls into this category, and is required for Appendix IX analysis. For this analyte a mass chromatogram search is made.

12.2.1. Hexachlorophene

Display the mass chromatograms for mass 196 and mass 198 for the region of the chromatogram from at least 2 minutes before chrysene-d12 to at least 4 minutes after chrysene-d12. If peaks for both ions coincide then the analyst evaluates the spectrum for the presence of hexachlorophene. No quantitation is possible.

12.3. For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the type of analyses being conducted. Computer generated library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual comparison of sample spectra with the nearest library searches shall the mass spectral interpretation specialist assign a tentative identification. Guidelines for making tentative identification are:

- Relative intensities of major ions in the reference spectrum (ions >10% of the most abundant ion) should be present in the sample spectrum.
- The relative intensities of the major ions should agree within ±20%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance should be between 30%and 70%.)
- Molecular ions present in the reference spectrum should be present in the sample spectrum.
- Ions present in the sample spectrum, but not in the reference spectrum, should be reviewed for possible background contamination or presence of coeluting compounds.

- Ions present in the reference spectrum, but not in the sample spectrum, should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting peaks. Data system library reduction programs can sometimes create these discrepancies.
- Automatic background subtraction can severely distort spectra from samples with unresolved hydrocarbons.

12.4. Anyone evaluating data is trained to know how to handle isomers with identical mass spectra and close elution times. These include:

Dichlorobenzenes
Methylphenols
Trichlorophenols
Phenanthrene, anthracene
Fluoranthene, pyrene
Benzo(b) and (k)fluoranthene
Chrysene, benzo(a)anthracene

Extra precautions concerning these compounds are to more closely scrutinize retention time vs. the calibration standard and also to check that all isomers have distinct retention times.

A second category of problem compounds would be the poor responders or compounds that chromatograph poorly. Included in this category would be:

Benzoic acid
Chloroanilines
Nitroanilines
2,4-Dinitrophenol
4-Nitrophenol
Pentachlorophenol
3,3'-Dichlorobenzidine
Benzyl alcohol
4,6-Dinitro-2-methylphenol

Manually checking the integrations would be appropriate for these compounds.

12.5. Calculations

12.5.1. Percent Relative Standard Deviation for Initial Calibration

$$\%RSD = \frac{SD}{\overline{RF}} \times 100$$

$\overline{RF}$ = Mean of RFs from intial caibration for a compound

SD = Standard deviation of RFs from initial calibration for a compound,

$$= \sqrt{\sum_{i=1}^{N} \frac{\left(RFi - \overline{RF}\right)^2}{N-1}}$$

RFi = RF for each of the calibration levels

N = Number of RF values

12.5.2. Continuing Calibration Percent Difference

$$\%Difference = \frac{RFAnalyte - \overline{RF}}{\overline{RF}} \times 100\%$$

12.5.3. Continuing calibration percent drift

$$\%\,Drift = \frac{C_{actual} - C_{found}}{C_{actual}} \times 100\%$$

C_{actual} = Known concentration in standard

C_{found} = Measured concentration using selected quantitation method

12.5.4. Concentration in the extract

The concentration of each identified analyte and surrogate in the extract is calculated from the linear or quadratic curve fitted to the initial calibration points, or from the average RF of the initial calibration.

12.5.4.1. Average response factor

If the average of all the %RSDs of the response factors in the initial calibration is ≤ 15%, the average response factor from the initial calibration may be used for quantitation.

$$C_{ex} = \frac{R_x C_{is}}{R_{is}\overline{RF}}$$

12.5.4.2. Linear fit

$$C_{ex} = A + B\frac{(R_x C_{is})}{R_{is}}$$

C_{ex} = Concentration in extract, µg/ml
R_x = Response for analyte
R_{is} = Response for internal standard
C_{is} = Concentration of internal standard
A = Intercept
B = Slope

12.5.4.3. Quadratic fit

$$C_{ex} = A + B\left(\frac{R_x C_{is}}{R_{is}}\right) + C\left(\frac{R_x C_{is}}{R_{is}}\right)$$

C = Curvature

12.5.5. The concentration in the sample is then calculated.

12.5.5.1. Aqueous Calculation

$$\text{Concentration, } \mu g / L = \frac{C_{ex} V_t}{V_o}$$

Where:

V_t = Volume of total extract, µL, taking into account dilutions (i.e., a 1-to-10 dilution of a 1 ml extract will mean V_t = 10,000 µL. If half of the base/neutral extract and half of the acid extract are combined, V_t = 2,000.)

V_o = Volume of water extracted (ml)

12.5.6. Sediment/Soil, Sludge (on a dry-weight basis) and Waste (normally on a wet-weight basis:

$$\text{Concentration, } \mu g / kg = \frac{C_{ex} V_t}{W_s D}$$

W_s = Weight of sample extracted or diluted in grams
D = (100 - % moisture in sample)/100, for a dry weight basis or 1 for a wet weight basis

12.6. MS/MSD percent recovery calculation.

$$\text{Matrix Spike Recovery} = \frac{S_{SR} - S_R}{S_A} \times 100\%$$

S_{SR} = Spike sample result
S_R = Sample result
S_A = Spike added

12.7. Relative % Difference calculation for the MS/MSD

$$RPD = \frac{MS_R - MSD_R}{1/2(MS_R + MSD_R)} \times 100$$

RPD = Relative percent difference
MS_R = Matrix spike result
MSD_R = Matrix spike duplicate result

12.8. Relative response factor calculation.

$$RF = \frac{A_x C_{is}}{A_{is} C_x}$$

A_x=Area of the characteristic ion for the compound being measured
A_{is}=Area of the characteristic ion for the specific internal standard
C_x=Concentration of the compound being measured (µg/L)
C_{is} =Concentration of the specific internal standard (µg/L)

12.9. Calculation of TICs: The calculation of TICs (tentatively identified compounds) is identical to the above calculations with the following exceptions:

A_x= Area of the total ion chromatogram for the compound being measured
A_{is}= Area of the total ion chromatogram for the nearest internal standard without interference
RF= 1

13. DATA ASSESSMENT AND ACCEPTANCE CRITERIA

The following represent data assessment for samples and acceptance criteria for QC measures. Corrective actions for any failure in QC measures are shown in Section 14.

13.1. QC sample acceptance criteria

13.1.1. Method Blank.

13.1.1.1. No target analytes may be present in the method blank above the reporting limit with the exception of the common laboratory solvents methylene chloride and acetone.

13.1.1.2. The method blank must have acceptable surrogate recoveries.

13.1.2. Laboratory Control Sample (LCS).

13.1.2.1. All control analytes must be within established control limits for accuracy (%Recovery) and precision (RPD). Exceptions are allowed only with QA and project management approval.

13.1.2.2. It is desirable to have all non-control analytes within control limits, but it is expected that a small proportion will not be in control.

13.1.2.3. The LCS must have acceptable surrogate recoveries.

13.1.3. Matrix Spike/Matrix Spike Duplicate (MS/MSD).

13.1.3.1. All analytes should be within established control limits for accuracy (%Recovery) and precision (RPD). Deviations from this may be the results of matrix effects, which are confirmed by passing LCSs.

13.1.3.2. No specific corrective actions are required in the evaluation of the MS/MSD results provided that the batch LCS is in control. Analysts should use sound judgment in accepting MS/MSD results that are not within control limits, especially if the LCS results are borderline.

13.2. Sample result evaluation

13.2.1. Carryover. When a sample has a high response for a compound, there is a real possibility that some of the sample may carry over into the sample analyzed immediately afterward.

13.2.1.1. If a sample analyzed after a sample with high concentrations has negative results, carryover did not occur.

13.2.1.2. If a sample analyzed after a sample with high concentrations has positive results for the same analytes, or if the chromatographic profile resembles the previous sample, the results are questionable. This sample must be reanalyzed under conditions in which carryover can be confirmed to not have occurred.

13.2.2. Dilutions

13.2.2.1. If the response for any compound exceeds the working range of the analytical system, a dilution of the extract is prepared and analyzed. An appropriate dilution should be in the upper half of the calibration range.

13.2.2.2. If the initial diluted run has no hits or hits below 20% of the calibration range and the matrix allows for analysis at a lesser dilution, then the sample must be reanalyzed at a dilution targeted to bring the largest hit in the upper half of the calibration range.

14. CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

14.1. Method Blank.

14.1.1. The samples in the batch associated to the defective method blank are evaluated.

14.1.1.1. If the analyte found in the method blank is confirmed to not be present in any of the associated samples at any level, the contamination did not affect those samples. This represents a non-impact situation and no specific corrective action is taken. A nonconformance memo is written to notify project management of the situation for evaluation against project requirements.

14.1.1.2. If the analyte found in the method blank is confirmed to be present in one or more of the associated samples, the concentrations are compared.

14.1.1.2.1. If the concentration in the method blank exceeded 10% of concentration found in one or more samples, the prescribed corrective action is to re-analyze all affected samples.

14.1.1.2.2. If the concentration in the method blank was less than 10% of the concentration found in one or more samples, the sample can be reported by qualifying the affected analytes. A nonconformance memo is written to notify project management of the situation for evaluation against project requirements.

14.1.2. If the analyte found in the method blank is not one of the common laboratory contaminants (Specify which), the analytical system must be placed out of service until the source of the problem is identified and corrected.

14.2. Laboratory control sample

14.2.1. If any control analyte is out of control for accuracy (% Recovery), the associated samples are evaluated.

14.2.1.1. If the recovery is biased high and the associated samples have no positive results for that analyte, a non-impact situation ensues. A nonconformance memo is written to notify project management of the situation for evaluation against project requirements.

14.2.1.2. If the recovery is biased low and the associated samples have positive results for that analyte, a minimal impact situation ensues. A nonconformance memo is written to notify project management of the situation for evaluation against project requirements.

14.2.1.3. If the recovery is biased high and the associated samples have positive results for that analyte, the prescribed corrective action is reanalysis of the affected sample(s).

14.2.1.4. If the recovery is biased low and the samples have no positive results for that analyte, the prescribed corrective action is reanalysis of the affected sample(s).

14.2.2. If any control analyte is out of control for precision (RPD), the associated samples are evaluated. All decisions about corrective action(s) are made in consultation with the project manager.

14.2.2.1. If there are no positive results in one or more samples, a non-impact situation ensues for those samples. A nonconformance memo is written to notify project management of the situation for evaluation against project requirements.

14.2.2.2. If there are positive results for one or more analytes, the likelihood of poor reproducibility increases and corrective action must be evaluated. A nonconformance memo is written to notify project management of the situation for a project decision on whether the affected sample(s) should be reanalyzed.

15. CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

15.1. Method blanks

15.1.1. If there is insufficient sample to perform re-analysis, the analyst must notify the project manager for consultation with the client. In this situation, all associated samples are flagged with a "B" qualifier and appropriate comments in the narrative.

15.2. LCS

15.2.1. If the batch is not re-extracted and reanalyzed, the reasons for accepting the batch must be clearly presented in the project records and the report. Acceptable matrix spike recoveries are not sufficient justification to preclude re-extraction of the batch.

15.2.2. If re-extraction and reanalysis of the batch is not possible due to limited sample volume or other constraints, the LCS is reported, all associated samples are flagged, and appropriate comments are made in a narrative to provide further documentation.

15.3. Insufficient sample

15.3.1. If there is insufficient sample to repeat the analysis, the project manager is notified via NCM for consultation with the client.

16. METHOD PERFORMANCE

16.1. Method Detection Limit

Each laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in QA Policy #: QA-005.

16.2. Initial Demonstration

Each laboratory must make an initial demonstration of capability for each individual method. Demonstration of capability for both soil and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest.

16.2.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to the level 4 calibration standard.

16.2.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these results with the acceptance criteria given in table 14.

16.2.3. If any analyte does not meet the acceptance criteria the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

16.3. Non-standard analytes

For non-standard analytes, an MDL study must be performed and calibration curve generated before analyzing any samples, unless lesser requirements are previously agreed to with the client. In any event, the minimum initial demonstration required is analysis of an extracted standard at the reporting limit and a single point calibration.

16.4. Training Qualification

The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

16.5. Data Quality Objectives (DQO). Refer to project-specific Quality Assurance plans for DQO information.

17. POLLUTION PREVENTION

This procedure will be carried out in a manner consistent with all applicable federal state and local regulations regarding pollution control and prevention. Specific controls due to the accidental release of hazardous materials can be found in the Chemical Hygiene Plan and facility attachments.

18. WASTE MANAGEMENT

Waste generated in this procedure will be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Coordinator should be contacted if additional information is required.

19. REFERENCES

19.1. SW846, Test Methods for Evaluating Solid Waste, Third Edition, Update III, December 1996, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS): Method 8270B.

19.2. J. W. Eichelberger, L. E. Harris, and W. L. Budde, "Reference Compound to Calibrate Ion Abundance Measurement in Gas Chromatography/Mass Spectrometry," Analytical Chemistry, 47, 995 (1975)

19.3. 40CFR Part 136: "Guidelines Establishing Test Procedures for the Analysis of Pollutants, Appendix A, "Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater", Code of Federal Regulations, Revised July1, 1995, Method 625 "Base/Neutrals and Acids".

20. CLARIFICATIONS, MODIFICATIONS AND ADDITIONS TO REFERENCE METHOD

20.1. Modifications from Reference Method

20.1.1. A retention time window of 0.2 minutes is used for all components, since some data systems do not have the capability of using the relative retention time units specified in the reference method.

20.1.2. The quantitation and qualifier ions for some compounds have been changed from those recommended in SW-846 in order to improve the reliability of qualitative identification.

20.2. Modifications from Previous Revision

20.2.1. This SOP has been substantially revised to meet the requirements of method 8270C.

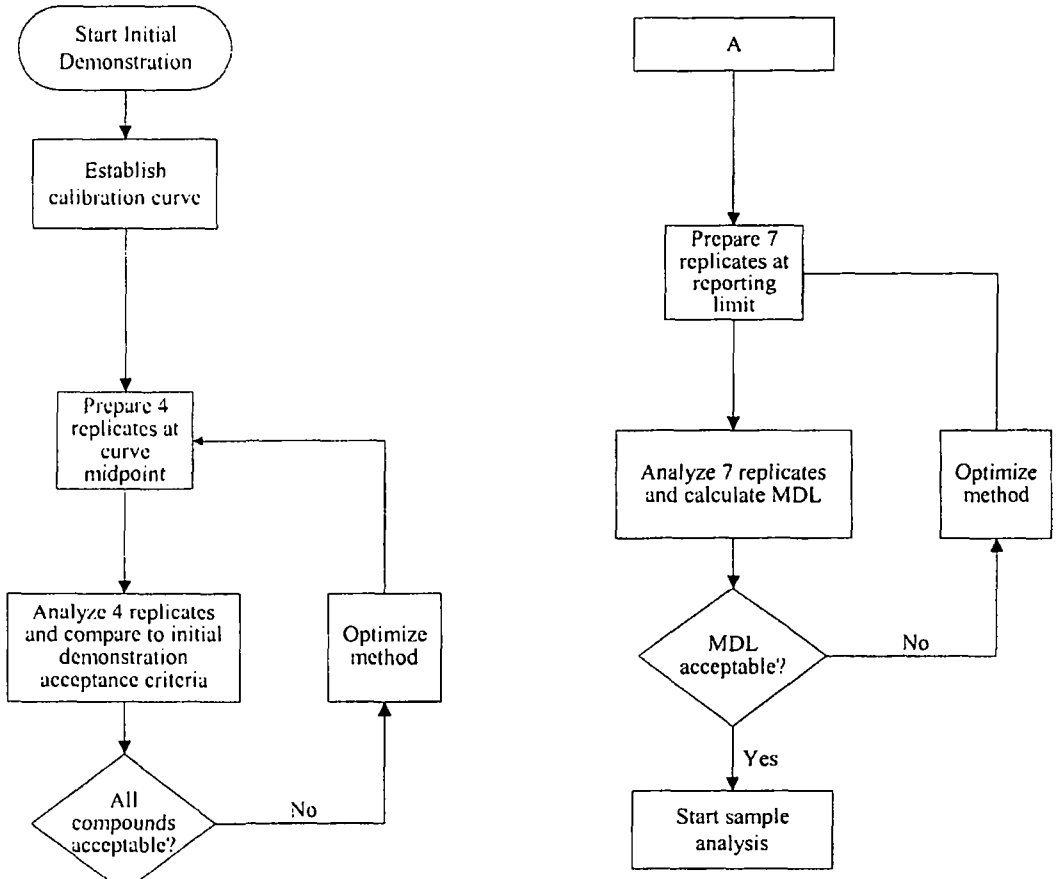
20.2.2. Directions for analysis be method 625 have been added as an attachment.

20.3. Facility Specific SOPs

This SOP has been revised to create a facility-specific version of the Corporate SOP, no facility-specific attachments to the SOP are required.

20.4. Flow Diagrams and Tables

20.4.1. Initial Demonstration and MDL


Start Initial Demonstration
Establish calibration curve
Prepare 4 replicates at curve midpoint
Analyze 4 replicates and compare to initial demonstration acceptance criteria
Optimize method
All compounds acceptable?
No
Yes
A
A
Prepare 7 replicates at reporting limit
Analyze 7 replicates and calculate MDL
Optimize method
MDL acceptable?
No
Yes
Start sample analysis

20.4.2. Tables

Table 1
STL Primary Standard[1] and Standard Reporting Limits

Analytes	CAS Number	Standard Reporting Limits	
		Aqueous µg/L	Low Soil/Sediment µg/kg
Pyridine	110-86-1	20	660
N-nitrosodimethylamine	62-75-9	10	330
Aniline	62-53-3	10	330
Phenol	108-95-2	10	330
Bis(2-chloroethyl)ether	111-44-4	10	330
2-Chlorophenol	95-57-8	10	330
1,3-Dichlorobenzene	541-73-1	10	330
1,4-Dichlorobenzene	106-46-7	10	330
Benzyl alcohol	100-51-6	10	330
1,2-Dichlorobenzene	95-50-1	10	330
2-Methylphenol	95-48-7	10	330
2,2'-oxybis(1-chloropropane)[2]	108-60-1	10	330
4-Methylphenol	106-44-5	10	330
N-Nitroso-di-n-propylamine	621-64-7	10	330
Hexachloroethane	67-72-1	10	330
Nitrobenzene	98-95-3	10	330
Isophorone	78-59-1	10	330
2-Nitrophenol	88-75-5	10	330
2,4-Dimethylphenol	105-67-9	10	330
Benzoic acid	65-85-0	50	1600
Bis(2-chloroethoxy)methane	111-91-1	10	330
2,4-Dichlorophenol	120-83-2	10	330
1,2,4-Trichlorobenzene	120-82-1	10	330
Naphthalene	91-20-3	10	330
4-Chloroaniline	106-47-8	10	330
Hexachlorobutadiene	87-68-3	10	330
4-Chloro-3-methylphenol	59-50-7	10	330
2-Methylnaphthalene	91-57-6	10	330
Hexachlorocyclopentadiene	77-47-4	50	1600
2,4,6-Trichlorophenol	88-06-2	10	330
2,4,5-Trichlorophenol	95-95-4	10	330
2-Chloronaphthalene	91-58-7	10	330
2-Nitroaniline	88-74-4	50	1600
Dimethyl phthalate	131-11-3	10	330
Acenaphthylene	208-96-8	10	330
3-Nitroaniline	99-09-2	50	1600
Acenaphthene	83-32-9	10	330
2,4-Dinitrophenol	51-28-5	50	1600
4-Nitrophenol	100-02-7	50	1600
Dibenzofuran	132-64-9	10	330
2,4-Dinitrotoluene	121-14-2	10	330
2,6-Dinitrotoluene	606-20-2	10	330
Diethylphthalate	84-66-2	10	330

Table 1
STL Primary Standard[1] and Standard Reporting Limits

Analytes	CAS Number	Standard Reporting Limits	
		Aqueous µg/L	Low Soil/Sediment µg/kg
4-Chlorophenyl phenyl ether	7005-72-3	10	330
Fluorene	86-73-7	10	330
4-Nitroaniline	100-01-6	50	1600
4,6-Dinitro-2-methylphenol	534-52-1	50	1600
N-Nitrosodiphenylamine	86-30-6	10	330
Azobenzene	103-33-3	10	330
4-Bromophenyl phenyl ether	101-55-3	10	330
Hexachlorobenzene	118-74-1	10	330
Pentachlorophenol	87-86-5	50	1600
Phenanthrene	85-01-8	10	330
Anthracene	120-12-7	10	330
Carbazole	86-74-8	10	330
Di-n-butyl phthalate	84-74-2	10	330
Fluoranthene	206-44-0	10	330
Benzidine	92-87-5	100	3300
Pyrene	129-00-0	10	330
Butyl benzyl phthalate	85-68-7	10	330
3,3'-Dichlorobenzidine	91-94-1	50	1600
Benzo(a)anthracene	56-55-3	10	330
Bis(2-ethylhexyl)phthalate	117-81-7	10	330
Chrysene	218-01-9	10	330
Di-n-octylphthalate	117-84-0	10	330
Benzo(b)fluoranthene	205-99-2	10	330
Benzo(k)fluoranthene	207-08-9	10	330
Benzo(a)pyrene	50-32-8	10	330
Indeno(1,2,3-cd)pyrene	193-39-5	10	330
Dibenz(a,h)anthracene	53-70-3	10	330
Benzo(g,h,i)perylene	191-24-2	10	330

[1] The STL primary standard is the standard normally used at STL. Additional standards, such as the Appendix IX standard may be necessary to include all target analytes required for some clients.

2,2'oxybis(1-chloropropane) was formally known as bis(2-chloroisopropyl)ether

Table 2
STL Appendix IX[1] Standard Reporting Limits

Semivolatiles	CAS Number	Standard Reporting Limits	
		Aqueous µg/L	Low Soil/Sediment µg/kg
2-Picoline	109-06-8	20	660
N-Nitrosomethylethylamine	10595-95-6	10	330
Methyl methanesulfonate	66-27-3	10	330
N-Nitrosodiethylamine	55-18-5	10	330
Ethyl methanesulfonate	62-50-0	10	330
Pentachloroethane	76-01-7	50	1600
Acetophenone	98-86-2	10	330
N-Nitrosopyrrolidine	930-55-2	10	330
N-Nitrosomorpholine	59-89-2	10	330
o-Toluidine	95-53-4	20	660
3-Methylphenol	108-39-4	10	330
N-Nitrosopiperidine	100-75-4	10	330
o,o,o-Triethyl-Phosphorothioate[2]	126-68-1	50	1600
a,a-Dimethyl-phenethylamine	122-09-8	50	1600
2,6-Dichlorophenol	87-65-0	10	330
Hexachloropropene	1888-71-7	100	3300
p-Phenylenediamine	106-50-3	100	3300
n-Nitrosodi-n-butylamine	924-16-3	10	330
Safrole	94-59-7	20	660
1,2,4,5-Tetrachlorobenzene	95-94-3	10	330
Isosafrole	120-58-1	20	660
1,4-Dinitrobenzene	100-25-4	10	330
1,4-Naphthoquinone	130-15-4	50	1600
1,3-Dinitrobenzene	99-65-0	10	330
Pentachlorobenzene	608-93-5	10	330
1-Naphthylamine	134-32-7	10	330
2-Naphthylamine	91-59-8	10	330
2,3,4,6-Tetrachlorophenol	58-90-2	50	1600
5-Nitro-o-toluidine	99-55-8	20	660
Thionazin[2]	297-97-2	50	1600
1,3,5-Trinitrobenzene	99-35-4	50	1600
Sulfotepp[2]	3689-24-5	50	1600
Phorate[2]	298-02-2	50	1600
Phenacetin	62-44-2	20	660
Diallate[3]	2303-16-4	20	660
Dimethoate[2]	60-51-5	20	660
4-Aminobiphenyl	92-67-1	50	1600
Pentachloronitrobenzene	82-68-8	50	1600
Pronamide	23950-58-5	20	660
Disulfoton[2]	298-04-4	50	1600
2-secbutyl-4,6-dinitrophenol (Dinoseb)	88-85-7	20	660
Methyl Parathion[2]	298-00-0	50	1600
4-Nitroquinoline-1-oxide	56-57-5	100	3300

Table 2
STL Appendix IX[1] Standard Reporting Limits

Semivolatiles	CAS Number	Standard Reporting Limits	
		Aqueous µg/L	Low Soil/Sediment µg/kg
Parathion[2]	56-38-2	50	1600
Methapyrilene	91-80-5	50	1600
Aramite	140-57-8	20	660
Isodrin[3]	465-73-6	10	330
Kepone[2]	143-50-0	100	3300
Famphur[3]	52-85-7	100	3300
p-(Dimethylamino)azobenzene	60-11-7	20	660
p-Chlorobenzilate[3]	510-15-6	10	330
3,3'-Dimethylbenzidine	119-93-7	50	1600
2-Acetylaminofluorene	53-96-3	100	3300
Dibenz(a,j)acridine	224-42-0	20	660
Hexachlorophene	70-30-4	100	3300
7,12-Dimethylbenz(a)anthracene	57-97-6	20	660
3-Methylcholanthrene	56-49-5	20	660

[1] The Appendix IX standard contains additional analytes required for the Appendix IX list. The STL primary standard must also be analyzed to include all of the Appendix IX list.
[2] May also be analyzed by method 8141, which can achieve lower reporting limits.
[3] May also be analyzed by method 8081, which can achieve lower reporting limits

Table 3
Reportable Analytes for STL Standard Tests, Primary Standard

Analyte	CAS Number	STL Standard List	TCLP	TCL	Appendix IX
Pyridine	110-86-1		X		X
N-nitrosodimethylamine	62-75-9				X
Aniline	62-53-3				X
Phenol	108-95-2	X		X	X
Bis(2-chloroethyl)ether	111-44-4	X		X	X
2-Chlorophenol	95-57-8	X		X	X
1,3-Dichlorobenzene	541-73-1	X		X	X
1,4-Dichlorobenzene	106-46-7	X	X	X	X
Benzyl alcohol	100-51-6				X
1,2-Dichlorobenzene	95-50-1	X		X	X
2-Methylphenol	95-48-7	X	X	X	X
2,2'-oxybis(1-chloropropane)[1]	180-60-1	X		X	X
4-Methylphenol	106-44-5	X	X	X	X
N-Nitroso-di-n-propylamine	621-64-7	X		X	X
Hexachloroethane	67-72-1	X	X	X	X
Nitrobenzene	98-95-3	X	X	X	X
Isophorone	78-59-1	X		X	X
2-Nitrophenol	88-75-5	X		X	X
2,4-Dimethylphenol	105-67-9	X		X	X
Benzoic acid	65-85-0				
Bis(2-chloroethoxy)methane	111-91-1	X		X	X
2,4-Dichlorophenol	120-83-2	X		X	X
1,2,4-Trichlorobenzene	120-82-1	X		X	X
Naphthalene	91-20-3	X		X	X
4-Chloroaniline	106-47-8	X		X	X
Hexachlorobutadiene	87-68-3	X	X	X	X
4-Chloro-3-methylphenol	59-50-7	X		X	X
2-Methylnaphthalene	91-57-6	X		X	X
Hexachlorocyclopentadiene	77-47-4	X		X	X
2,4,6-Trichlorophenol	88-06-2	X	X	X	X
2,4,5-Trichlorophenol	95-95-4	X	X	X	X
2-Chloronaphthalene	91-58-7	X		X	X
2-Nitroaniline	88-74-4	X		X	X
Dimethyl phthalate	131-11-3	X		X	X
Acenaphthylene	208-96-8	X		X	X
3-Nitroaniline	99-09-2	X		X	X
Acenaphthene	83-32-9	X		X	X
2,4-Dinitrophenol	51-28-5	X		X	X
4-Nitrophenol	100-02-7	X		X	X
Dibenzofuran	132-64-9	X		X	X
2,4-Dinitrotoluene	121-14-2	X	X	X	X
2,6-Dinitrotoluene	606-20-2	X		X	X
Diethylphthalate	84-66-2	X		X	X
4-Chlorophenyl phenyl ether	7005-72-3	X		X	X

Table 3
Reportable Analytes for STL Standard Tests, Primary Standard

Analyte	CAS Number	STL Standard List	TCLP	TCL	Appendix IX
Fluorene	86-73-7	X		X	X
4-Nitroaniline	100-01-6	X		X	X
4,6-Dinitro-2-methylphenol	534-52-1	X		X	X
N-Nitrosodiphenylamine	86-30-6	X		X	X
Azobenzene[4]	103-33-3				
4-Bromophenyl phenyl ether	101-55-3	X		X	X
Hexachlorobenzene	118-74-1	X	X	X	X
Pentachlorophenol	87-86-5	X	X	X	X
Phenanthrene	85-01-8	X		X	X
Anthracene	120-12-7	X		X	X
Carbazole	86-74-8	X		X	
Di-n-butyl phthalate	84-74-2	X		X	X
Fluoranthene	206-44-0	X		X	X
Benzidine	92-87-5				
Pyrene	129-00-0	X		X	X
Butyl benzyl phthalate	85-68-7	X		X	X
3,3'-Dichlorobenzidine	91-94-1	X		X	X
Benzo(a)anthracene	56-55-3	X		X	X
Bis(2-ethylhexyl)phthalate	117-81-7	X		X	X
Chrysene	218-01-9	X		X	X
Di-n-octylphthalate	117-84-0	X		X	X
Benzo(b)fluoranthene	205-99-2	X		X	X
Benzo(k)fluoranthene	207-08-9	X		X	X
Benzo(a)pyrene	50-32-8	X		X	X
Indeno(1,2,3-cd)pyrene	193-39-5	X		X	X
Dibenz(a,h)anthracene	53-70-3	X		X	X
Benzo(g,h,i)perylene	191-24-2	X		X	X

[1] 2,2'oxybis(1-chloropropane) was formally known as bis(2-chloroisopropyl)ether

[2] Azobenzene is formed by decomposition of 1,2-diphenlyhydrazine. If 1,2-diphenylhydrazine is requested, it will be analyzed as azobenzene.

Table 4
Reportable analytes for STL Standard Tests, Appendix IX Standard

Semivolatiles	CAS Number	STL Standard List	TCLP	TCL	Appendix IX
2-Picoline	109-06-8				X
N-Nitrosomethylethylamine	10595-95-6				X
Methyl methanesulfonate	66-27-3				X
N-Nitrosodiethylamine	55-18-5				X
Ethyl methanesulfonate	62-50-0				X
Pentachloroethane	76-01-7				X
Acetophenone	98-86-2				X
N-Nitrosopyrrolidine	930-55-2				X
N-Nitrosomorpholine	59-89-2				X
o-Toluidine	95-53-4				X
3-Methylphenol	108-39-4				X
N-Nitrosopiperidine	100-75-4				X
o,o,o-Triethyl-Phosphorothioate[2]	126-68-1				X
a,a-Dimethyl-phenethylamine	*122-09-8*				X
2,6-Dichlorophenol	87-65-0				X
Hexachloropropene	1888-71-7				X
p-Phenylenediamine	106-50-3				X
n-Nitrosodi-n-butylamine	924-16-3				X
Safrole	94-59-7				X
1,2,4,5-Tetrachlorobenzene	95-94-3				X
Isosafrole	120-58-1				X
1,4-Dinitrobenzene	100-25-4				
1,4-Naphthoquinone	130-15-4				X
1,3-Dinitrobenzene	99-65-0				X
Pentachlorobenzene	608-93-5				X
1-Naphthylamine	134-32-7				X
2-Naphthylamine	91-59-8				X
2,3,4,6-Tetrachlorophenol	58-90-2				X
5-Nitro-o-toluidine	99-55-8				X
Thionazin[2]	297-97-2				X
1,3,5-Trinitrobenzene	99-35-4				X
Sulfotepp[2]	3689-24-5				X
Phorate[2]	298-02-2				X
Phenacetin	62-44-2				X
Diallate	2303-16-4				X
Dimethoate[2]	60-51-5				X
4-Aminobiphenyl	92-67-1				X
Pentachloronitrobenzene	82-68-8				X
Pronamide	23950-58-5				X
Disulfoton[2]	298-04-4				X
2-secbutyl-4,6-dinitrophenol (Dinoseb)[2]	88-85-7				X
Methyl parathion[2]	298-00-0				X
4-Nitroquinoline-1-oxide	56-57-5				X

Table 4
Reportable analytes for STL Standard Tests, Appendix IX Standard

Semivolatiles	CAS Number	STL Standard List	TCLP	TCL	Appendix IX
Parathion[2]	56-38-2				X
Isodrin[3]	465-73-6				X
Kepone [2]	143-50-0				X
Famphur[2]	52-85-7				X
Methapyrilene	91-80-5				X
Aramite	140-57-8				X
p-(Dimethylamino)azobenzene	60-11-7				X
p-Chlorobenzilate[3]	510-15-6				X
3,3'-Dimethylbenzidine	119-93-7				X
2-Acetylaminofluorene	53-96-3				X
Dibenz(a,j)acridine	224-42-0				
7,12-Dimethylbenz(a)anthracene	57-97-6				X
3-Methylcholanthrene	56-49-5				X
Hexachlorophene[4]	70-30-4				X
Diphenylamine[5]	122-39-4				X

2 May also be analyzed by method 8141, which can achieve lower reporting limits.
3 May also be analyzed by method 8081, which can achieve lower reporting limits
4 Diphenylamine is a required compound for Appendix IX. N-nitrosodiphenylamine decomposes in the injection port to form diphenylamine. Therefore these two compounds cannot be distinguished. Diphenylamine is not included in the calibration standard.

Table 5
Suggested Instrumental Conditions

Mass Range	35-500 amu
Scan Time	≤1 second/scan
Initial Column Temperature/Hold Time	40°C for 2 minutes
Column Temperature Program	40 - 320°C at 11.5°C/min
Final Column Temperature/Hold Time	320°C (until at least one minute after benzo(g,h,i)perylene has eluted)
Injector Temperature	250 - 300°C
Transfer Line Temperature	250 - 300°C
Source Temperature	According to manufacturer's specifications
Injector	Grob-type, split / splitless
Sample Volume	1 or 2 µl
Carrier Gas	Helium at 30 cm/sec

Table 6
DFTPP Key Ions and Ion Abundance Criteria

Mass	Ion Abundance Criteria
51	30 - 60% of mass 198
68	<2% of mass 69
70	<2% of mass 69
127	40 - 60% of mass 198
197	<1% of mass 198
198	Base peak, 100% relative abundance
199	5 - 9% of mass 198
275	10 - 30% of mass 198
365	>1% of mass 198
441	Present, but less than mass 443
442	>40% of mass 198
443	17 - 23% of mass 442

Table 7

Analytes in Approximate Retention Time Order and Characteristic Ions, Primary Standard

Analyte	Primary	Secondary	Tertiary
N-nitrosodimethylamine	74	42	
Pyridine	79	52	
2-Fluorophenol (Surrogate Standard)	112	64	63
Phenol-d5 (Surrogate Standard)	99	42	71
Aniline	93	66	
Phenol	94	65	66
Bis(2-chloroethyl)ether	93	63	95
2-Chlorophenol	128	64	130
1,3-Dichlorobenzene	146	148	113
1,4-Dichlorobenzene-d4 (Internal Standard)	152	150	115
1,4-Dichlorobenzene	146	148	113
Benzyl Alcohol	108	79	77
1,2-Dichlorobenzene	146	148	113
2-Methylphenol	108	107	79
2,2'-oxybis(1-chloropropane)[1]	45	77	79
4-Methylphenol	108	107	79
N-Nitroso-di-n-propylamine	70	42	101,130
Hexachloroethane	117	201	199
Nitrobenzene-d5 (Surrogate Standard)	82	128	54
Nitrobenzene	77	123	65
Isophorone	82	95	138
2-Nitrophenol	139	65	109
2,4-Dimethylphenol	107	121	122
Benzoic Acid	122	105	77
Bis(2-chloroethoxy)methane	93	95	123
2,4-Dichlorophenol	162	164	98
1,2,4-Trichlorobenzene	180	182	145
Naphthalene-d8 (Internal Standard)	136	68	54
Naphthalene	128	129	127
4-Chloroaniline	127	129	65
Hexachlorobutadiene	225	223	227
4-Chloro-3-methylphenol	107	144	142
2-Methylnaphthalene	142	141	115
Hexachlorocyclopentadiene	237	235	272
2,4,6-Trichlorophenol	196	198	200
2,4,5-Trichlorophenol	196	198	200
2-Fluorobiphenyl (Surrogate Standard)	172	171	170
2-Chloronaphthalene	162	164	127
2-Nitroaniline	65	92	138
Dimethylphthalate	163	194	164
Acenaphthylene	152	151	153

Table 7

Analytes in Approximate Retention Time Order and Characteristic Ions, Primary Standard

Analyte	Primary	Secondary	Tertiary
2,6-Dinitrotoluene	165	63	89
Acenaphthene-d10 (Internal Standard)	164	162	160
3-Nitroaniline	138	108	92
Acenaphthene	153	152	154
2,4-Dinitrophenol	184	63	154
Dibenzofuran	168	139	84
4-Nitrophenol	109	139	65
2,4-Dinitrotoluene	165	63	89
Diethylphthalate	149	177	150
Fluorene	166	165	167
4-Chlorophenylphenylether	204	206	141
4-Nitroaniline	138	92	108
4,6-Dinitro-2-methylphenol	198	182	77
N-Nitrosodiphenylamine	169	168	167
2,4,6-Tribromophenol (Surrogate Standard)	330	332	141
Azobenzene	77	182	105
4-Bromophenylphenylether	248	250	141
Hexachlorobenzene	284	142	249
Pentachlorophenol	266	264	268
Phenanthrene-d10 (Internal Standard)	188	94	80
Phenanthrene	178	179	176
Anthracene	178	179	176
Carbazole	167	166	139
Di-n-butylphthalate	149	150	104
Fluoranthene	202	101	100
Benzidine	184	92	185
Pyrene	202	101	100
Terphenyl-d14 (Surrogate Standard)	244	122	212
Butylbenzylphthalate	149	91	206
Benzo(a)Anthracene	228	229	226
Chrysene-d12 (Internal Standard)	240	120	236
3,3'-Dichlorobenzidine	252	254	126
Chrysene	228	226	229
Bis(2-ethylhexyl)phthalate	149	167	279
Di-n-octylphthalate	149	167	43
Benzo(b)fluoranthene	252	253	125
Benzo(k)fluoranthene	252	253	125
Benzo(a)pyrene	252	253	125
Perylene-d12 (Internal Standard)	264	260	265
Indeno(1,2,3-cd)pyrene	276	138	277
Dibenz(a,h)anthracene	278	139	279
Benzo(g,h,i)perylene	276	138	277

Table 8

Analytes in Approximate Retention Time Order and Characteristic Ions, Appendix IX Standard

Analyte	Primary	Secondary	Tertiary
2-Picoline	93	66	92
N-Nitrosomethylethylamine	88	42	43
Methyl methanesulfonate	80	79	65
N-Nitrosodiethylamine	102	44	57
Ethyl methanesulfonate	79	109	97
Pentachloroethane	117	119	167
Acetophenone	105	77	120
N-Nitrosopyrrolidine	100	41	42
N-Nitrosomorpholine	116	56	86
o-Toluidine	106	107	
3-Methylphenol	108	107	77
N-Nitrosopiperidine	114	42	55
o,o,o-Triethyl-Phosphorothioate	198	121	93
a,a-Dimethyl-phenethylamine	58	91	
2,6-Dichlorophenol	162	164	63
Hexachloropropene	213	215	211
p-Phenylenediamine	108	80	
n-Nitrosodi-n-butylamine	84	57	41
Safrole	162	104	77
1,2,4,5-Tetrachlorobenzene	216	214	218
Isosafrole 1	162	104	131
Isosafrole 2	162	104	131
1,4-Dinitrobenzene	168	75	122
1,4-Naphthoquinone	158	104	102
1,3-Dinitrobenzene	168	75	76
Pentachlorobenzene	250	248	252
1-Naphthylamine	143	115	
2-Naphthylamine	143	115	
2,3,4,6-Tetrachlorophenol	232	230	131
5-Nitro-o-toluidine	152	77	106
Thionazin	97	96	143
1,3,5-Trinitrobenzene	213	75	120
Sulfotepp	97	322	202
Phorate	75	97	121
Phenacetin	108	179	109
Diallate	86	234	
Dimethoate	87	93	125
4-Aminobiphenyl	169		
Pentachloronitrobenzene	237	142	214
Pronamide	173	175	255
Disulfoton	88	97	89
2-secbutyl-4,6-dinitrophenol (Dinoseb)	211	163	147
Methyl parathion	109	125	263
4-Nitroquinoline-1-oxide	190	128	160

Table 8

Analytes in Approximate Retention Time Order and Characteristic Ions, Appendix IX Standard

Analyte	Primary	Secondary	Tertiary
Parathion	109	97	291
Isodrin	193	66	195
Kepone	272	274	237
Famphur	218	125	93
Methapyrilene	97	58	
Aramite 1	185	319	
Aramite 2	185	319	
p-(Dimethylamino)azobenzene	120	225	77
p-Chlorobenzilate	251	139	253
3,3'-Dimethylbenzidine	212	106	
2-Acetylaminofluorene	181	180	223
Hexachlorophene	196	198	
Dibenz(a,j)acridine	279	280	
7,12-Dimethylbenz(a)anthracene	256	241	120
3-Methylcholanthrene	268	252	253

Table 9

8270C LCS Compounds

LCS Compounds	Spiking Level, ng/µL in extract[1]
1,2,4-Trichlorobenzene	50
Acenaphthene	50
2,4-Dinitrotoluene	50
Pyrene	50
N-Nitroso-di-n-propylamine	50
1,4-Dichlorobenzene	50
Pentachlorophenol	75
Phenol	75
2-Chlorophenol	75
4-Chloro-3-methylphenol	75
4-Nitrophenol	75

Table 10

TCLP LCS Compounds

LCS Compounds	Spiking Level, ng/µL in extract[1]
1,4-Dichlorobenzene	50
2,4-Dinitrotoluene	50
Hexachlorobenzene	50
Hexachlorobutadiene	50
Hexachloroethane	50
2-Methylphenol	50
3-Methylphenol	50
4-Methylphenol	50
Nitrobenzene	50
Pentachlorophenol	50
Pyridine	50
2,4,5-Trichlorophenol	50
2,4,6-Trichlorophenol	50

Recovery limits for the LCS and for matrix spikes are generated from historical data and are maintained by the QA department.

Table 11
8270C Surrogate Compounds

Surrogate Compounds	Spiking Level, ng/µL in extract[2]
Nitrobenzene-d5	50
2-Fluorobiphenyl	50
Terphenyl-d14	50
1,2-Dichlorobenzene-d4[1]	50
Phenol-d5	75
2-Fluorophenol	75
2,4,6-Tribromophenol	75
2-Chlorophenol-d4[1]	75

[1] Included in standard mix, but not routinely evaluated for method 8270C

Recovery limits for surrogates are generated from historical data and are maintained by the QA department.

Table 12
Calibration Levels, Primary Standard, µg/mL

Analyte	Level 1	Level 2	Level 3	Level 4	Level 5
Pyridine	10	25	40	60	80
N-nitrosodimethylamine	10	25	40	60	80
Aniline	10	25	40	60	80
Phenol	10	25	40	60	80
Bis(2-chloroethyl)ether	10	25	40	60	80
2-Chlorophenol	10	25	40	60	80
1,3-Dichlorobenzene	10	25	40	60	80
1,4-Dichlorobenzene	10	25	40	60	80
Benzyl alcohol	10	25	40	60	80
1,2-Dichlorobenzene	10	25	40	60	80
2-Methylphenol	10	25	40	60	80
2,2'-oxybis(1-chloropropane)[1]	10	25	40	60	80
4-Methylphenol	10	25	40	60	80
N-Nitroso-di-n-propylamine	10	25	40	60	80
Hexachloroethane	10	25	40	60	80
Nitrobenzene	10	25	40	60	80
Isophorone	10	25	40	60	80
2-Nitrophenol	10	25	40	60	80
2,4-Dimethylphenol	10	25	40	60	80
Benzoic acid	10	25	40	60	80
Bis(2-chloroethoxy)methane	10	25	40	60	80
2,4-Dichlorophenol	10	25	40	60	80
1,2,4-Trichlorobenzene	10	25	40	60	80
Naphthalene	10	25	40	60	80
4-Chloroaniline	10	25	40	60	80
Hexachlorobutadiene	10	25	40	60	80
4-Chloro-3-methylphenol	10	25	40	60	80
2-Methylnaphthalene	10	25	40	60	80
Hexachlorocyclopentadiene	10	25	40	60	80
2,4,6-Trichlorophenol	10	25	40	60	80
2,4,5-Trichlorophenol	10	25	40	60	80
2-Chloronaphthalene	10	25	40	60	80
2-Nitroaniline	10	25	40	60	80
Dimethyl phthalate	10	25	40	60	80
Acenaphthylene	10	25	40	60	80
3-Nitroaniline	10	25	40	60	80
Acenaphthene	10	25	40	60	80
2,4-Dinitrophenol	10	25	40	60	80
4-Nitrophenol	10	25	40	60	80
Dibenzofuran	10	25	40	60	80
2,4-Dinitrotoluene	10	25	40	60	80
2,6-Dinitrotoluene	10	25	40	60	80
Diethylphthalate	10	25	40	60	80
4-Chlorophenyl phenyl ether	10	25	40	60	80

Table 12
Calibration Levels, Primary Standard, µg/mL

Analyte	Level 1	Level 2	Level 3	Level 4	Level 5
Fluorene	10	25	40	60	80
4-Nitroaniline	10	25	40	60	80
4,6-Dinitro-2-methylphenol	10	25	40	60	80
N-Nitrosodiphenylamine	10	25	40	60	80
Azobenzene[2]	10	25	40	60	80
4-Bromophenyl phenyl ether	10	25	40	60	80
Hexachlorobenzene	10	25	40	60	80
Pentachlorophenol	10	25	40	60	80
Phenanthrene	10	25	40	60	80
Anthracene	10	25	40	60	80
Carbazole	10	25	40	60	80
Di-n-butyl phthalate	10	25	40	60	80
Fluoranthene	10	25	40	60	80
Benzidine	10	25	40	60	80
Pyrene	10	25	40	60	80
Butyl benzyl phthalate	10	25	40	60	80
3,3'-Dichlorobenzidine	10	25	40	60	80
Benzo(a)anthracene	10	25	40	60	80
Bis(2-ethylhexyl)phthalate	10	25	40	60	80
Chrysene	10	25	40	60	80
Di-n-octylphthalate	10	25	40	60	80
Benzo(b)fluoranthene	10	25	40	60	80
Benzo(k)fluoranthene	10	25	40	60	80
Benzo(a)pyrene	10	25	40	60	80
Indeno(1,2,3-cd)pyrene	10	25	40	60	80
Dibenz(a,h)anthracene	10	25	40	60	80
Benzo(g,h,i)perylene	10	25	40	60	80

[1] 2,2'oxybis(1-chloropropane) was formally known as bis(2-chloroisopropyl)ether
[2]Azobenzene is formed by decomposition of 1,2-diphenlyhydrazine. If 1,2-diphenylhydrazine is requested, it will be analyzed as azobenzene.

Table 13
Calibration Levels, Appendix IX Standard, µg/mL

Semivolatiles	Level 1	Level 2	Level 3	Level 4	Level 5
2-Picoline	10	25	40	60	80
N-Nitrosomethylethylamine	10	25	40	60	80
Methyl methanesulfonate	10	25	40	60	80
N-Nitrosodiethylamine	10	25	40	60	80
Ethyl methanesulfonate	10	25	40	60	80
Pentachloroethane	10	25	40	60	80
Acetophenone	10	25	40	60	80
N-Nitrosopyrrolidine	10	25	40	60	80
N-Nitrosomorpholine	10	25	40	60	80
o-Toluidine	10	25	40	60	80
3-Methylphenol	10	25	40	60	80
N-Nitrosopiperidine	10	25	40	60	80
o,o,o-Triethyl-Phosphorothioate	10	25	40	60	80
a,a-Dimethyl-phenethylamine	10	25	40	60	80
2,6-Dichlorophenol	10	25	40	60	80
Hexachloropropene	10	25	40	60	80
p-Phenylenediamine	100	250	400	600	800
n-Nitrosodi-n-butylamine	10	20	40	60	80
Safrole	10	20	40	60	80
1,2,4,5-Tetrachlorobenzene	10	20	40	60	80
Isosafrole 1 + 2	10	25	40	60	80
1,4-Dinitrobenzene	10	20	40	60	80
1,4-Naphthoquinone	10	20	40	60	80
1,3-Dinitrobenzene	10	20	40	60	80
Pentachlorobenzene	10	20	40	60	80
1-Naphthylamine	10	20	40	60	80
2-Naphthylamine	10	20	40	60	80
2,3,4,6-Tetrachlorophenol	10	20	40	60	80
5-Nitro-o-toluidine	10	20	40	60	80
Thionazin	10	20	40	60	80
1,3,5-Trinitrobenzene	50	125	200	300	400
Sulfotepp	10	25	40	60	80
Phorate	10	25	40	60	80
Phenacetin	10	25	40	60	80
Diallate 1 + 2	20	50	80	120	160
Dimethoate	10	25	40	60	80
4-Aminobiphenyl	10	25	40	60	80
Pentachloronitrobenzene	10	25	40	60	80
Pronamide	10	25	40	60	80
Disulfoton	10	25	40	60	80
2-secbutyl-4,6-dinitrophenol (Dinoseb)	10	25	40	60	80
Methyl parathion	10	25	40	60	80
4-Nitroquinoline-1-oxide	10	25	40	60	80
Parathion	10	25	40	60	80

Table 13
Calibration Levels, Appendix IX Standard, µg/mL

Semivolatiles	Level 1	Level 2	Level 3	Level 4	Level 5
Isodrin	10	20	50	100	80
Kepone	50	50	100	200	400
Famphur	50	50	100	200	400
Methapyrilene	10	20	50	100	80
Aramite 1 and 2	50	50	100	200	160
p-(Dimethylamino)azobenzene	10	20	50	100	80
p-Chlorobenzilate	10	20	50	100	80
3,3'-Dimethylbenzidine	10	20	50	100	80
Hexachlorophene	50	125	200	300	400
2-Acetylaminofluorene	10	20	50	100	80
Dibenz (a,j)acridine	10	20	50	100	80
7,12-Dimethylbenz(a)anthracene	10	20	50	100	80
3-Methylcholanthrene	10	20	50	100	80

Table 14
Initial demonstration recovery and precision limits

Compound	Spiking concentration µg/L	Limit for Relative Standard Deviation	Limit for average recovery, %
Acenaphthene	60	27.6	60.1-132.3
Acenaphthylene	60	40.2	53.5-126.0
Aldrin[1]	60	39.0	7.2-152.2
Anthracene	60	32.0	43.4-118.0
Benz(a)anthracene	60	27.6	41.8-133.0
Benzo(b)fluoranthene	60	38.8	42.0-140.4
Benzo(k)fluoranthene	60	32.3	25.2-145.7
Benzo(a)pyrene	60	39.0	31.7-148.0
Benzo(ghi)perylene	60	58.9	D-195.0
Benzylbutyl phthalate	60	23.4	D-139.9
B-BHC[1]	60	31.5	41.5-130.6
d-BHC[1]	60	21.6	D-100.0
Bis(2-chloroethyl) ether	60	55.0	42.9-126.0
Bis(2-chloroethoxy)methane	60	34.5	49.2-164.7
Bis(2-chloroisopropyl) ether	60	46.3	62.8-138.6
Bis(2-ethylhexyl) phthalate	60	41.1	28.9-136.8
4-Bromophenyl phenyl ether	60	23.0	64.9-114.4
2-Chloronaphthalene	60	13.0	64.5-113.5
4-Chlorophenyl phenyl ether	60	33.4	38.4-144.7
Chrysene	60	48.3	44.1-139.9
4,4'-DDD[1]	60	31.0	D-134.5
4,4'-DDE[1]	60	32.0	19.2-119.7
4,4'-DDT[1]	60	61.6	D-170.6
Dibenzo(a,h)anthracene	60	70.0	D-199.7
Di-n-butyl phthalate	60	16.7	8.4-111.0
1,2-Dichlorobenzene	60	30.9	48.6-112.0
1,3-Dichlorobenzene	60	41.7	16.7-153.9
1,4-Dichlorobenzene	60	32.1	37.3-105.7
3,3'-Dichlorobenzidine	60	71.4	8.2-212.5
Dieldrin[1]	60	30.7	44.3-119.3
Diethyl phthalate	60	26.5	D-100.0
Dimethyl phthalate	60	23.2	D-100.0
2,4-Dinitrotoluene	60	21.8	47.5-126.9
2,6-Dinitrotoluene	60	29.6	68.1-136.7
Di-n-octylphthalate	60	31.4	18.6-131.8
Endosulfan sulfate[1]	60	16.7	D-103.5
Endrin aldehyde	60	32.5	D-188.8
Fluoranthene	60	32.8	42.9-121.3
Fluorene	60	20.7	71.6-108.4
Heptachlor[1]	60	37.2	D-172.2
Heptachlor epoxide[1]	60	54.7	70.9-109.4

Table 14
Initial demonstration recovery and precision limits

Compound	Spiking concentration µg/L	Limit for Relative Standard Deviation	Limit for average recovery, %
Hexachlorobenzene	60	24.9	7.8-141.5
Hexachlorobutadiene	60	26.3	37.8-102.2
Hexachloroethane	60	24.5	55.2-100.0
Indeno(1,2,3-cd)pyrene	60	44.6	D-150.9
Isophorone	60	63.3	46.6-180.2
Naphthalene	60	30.1	35.6-119.6
Nitrobenzene	60	39.3	54.3-157.6
N-Nitrosodi-n-propylamine	60	55.4	13.6-197.9
PCB-1260[1]	60	54.2	19.3-121.0
Phenanthrene	60	20.6	65.2-108.7
Pyrene	60	25.2	69.6-100.0
1,2,4-Trichlorobenzene	60	28.1	57.3-129.2
4-Chloro-3-methylphenol	60	37.2	40.8-127.9
2-Chlorophenol	60	28.7	36.2-120.4
2,4-Chlorophenol	60	26.4	52.5-121.7
2,4-Dimethylphenol	60	26.1	41.8-109.0
2,4-Dinitrophenol	60	49.8	D-172.9
2-Methyl-4,6-dinitrophenol	60	93.2	53.0-100.0
2-Nitrophenol	60	35.2	45.0-166.7
4-Nitrophenol	60	47.2	13.0-106.5
Pentachlorophenol	60	48.9	38.1-151.8
Phenol	60	22.6	16.6-100.0
2,4,6-Trichlorophenol	60	31.7	52.4-129.2

[1]Since the organochlorine pesticides and PCBs are normally determined by method 8081/8082 at **STL**, they will not be included in the initial demonstration of capability for method 8270C.

ATTACHMENT A

MODIFICATIONS REQUIRED FOR ANALYSIS OF WASTEWATER FOLLOWING METHOD 625

21. REQUIREMENTS FOR METHOD 625

21.1. Method 625 is required for demonstration of compliance with NPDES wastewater discharge permits. The standard analyte list and reporting limits are listed in Table A-1.

21.2. This method can be applied only to aqueous matrices.

21.3. The tune period for this method is defined as 24 hours.

21.4. Initial calibration curve requirements:

21.4.1. The initial calibration curve for this method requires at least three points.

21.4.2. Target compounds must have RSD ≤ 35%.

21.4.3. If this requirement can not be met, a regression curve must be constructed for the non-compliant compounds.

21.5. Continuing calibration verification requirements: All target compounds must have %D ≤ 20%.

21.6. Matrix Spike and LCS requirements:

21.6.1. A full analyte spike is required for method 625. The spiking levels are given in Table A-2.

Table A-1. STL Method 625 standard reporting list and reporting limits.

Analytes	CAS Number	Aqueous µg/L
Phenol	*108-95-2*	*10*
Bis(2-chloroethyl)ether	111-44-4	10
2-Chlorophenol	95-57-8	10
1,3-Dichlorobenzene	541-73-1	10
1,4-Dichlorobenzene	106-46-7	10
1,2-Dichlorobenzene	95-50-1	10
2,2'-oxybis(1-chloropropane)	108-60-1	10
N-Nitroso-di-n-propylamine	621-64-7	10
Hexachloroethane	67-72-1	10
Nitrobenzene	98-95-3	10
Isophorone	78-59-1	10
2-Nitrophenol	88-75-5	10
2,4-Dimethylphenol	105-67-9	10
Bis(2-chloroethoxy)methane	111-91-1	10
2,4-Dichlorophenol	120-83-2	10
1,2,4-Trichlorobenzene	120-82-1	10
Naphthalene	91-20-3	10
Hexachlorobutadiene	87-68-3	10
4-Chloro-3-methylphenol	59-50-7	10
Hexachlorocyclopentadiene	77-47-4	50
2,4,6-Trichlorophenol	88-06-2	10
2-Chloronaphthalene	91-58-7	10
Dimethyl phthalate	131-11-3	10
Acenaphthylene	208-96-8	10
Acenaphthene	83-32-9	10
2,4-Dinitrophenol	51-28-5	50
4-Nitrophenol	100-02-7	50
2,4-Dinitrotoluene	121-14-2	10
2,6-Dinitrotoluene	606-20-2	10
Diethylphthalate	84-66-2	10
4-Chlorophenyl phenyl ether	7005-72-3	10
Fluorene	86-73-7	10
4,6-Dinitro-2-methylphenol	534-52-1	50
N-Nitrosodiphenylamine	86-30-6	10
4-Bromophenyl phenyl ether	101-55-3	10
Hexachlorobenzene	118-74-1	10
Pentachlorophenol	87-86-5	50
Phenanthrene	85-01-8	10
Anthracene	120-12-7	10
Di-n-butyl phthalate	84-74-2	10
Fluoranthene	206-44-0	10
Benzidine	92-87-5	100
Pyrene	129-00-0	10
Butyl benzyl phthalate	85-68-7	10
3,3'-Dichlorobenzidine	91-94-1	50
Benzo(a)anthracene	56-55-3	10
Bis(2-ethylhexyl)phthalate	117-81-7	10

Analytes	CAS Number	Aqueous µg/L
Chrysene	218-01-9	10
Di-n-octylphthalate	117-84-0	10
Benzo(b)fluoranthene	205-99-2	10
Benzo(k)fluoranthene	207-08-9	10
Benzo(a)pyrene	50-32-8	10
Indeno(1,2,3-cd)pyrene	193-39-5	10
Dibenz(a,h)anthracene	53-70-3	10
Benzo(g,h,i)perylene	191-24-2	10

Table A-2. Method 625 LCS and MS compounds and spike concentrations.

LCS Compounds	Spiking Level, ng/µL in extract[1]
Phenol	50
Bis(2-chloroethyl)ether	50
2-Chlorophenol	50
1,3-Dichlorobenzene	50
1,4-Dichlorobenzene	50
1,2-Dichlorobenzene	50
2,2'-oxybis(1-chloropropane)	50
N-Nitroso-di-n-propylamine	50
Hexachloroethane	50
Nitrobenzene	50
Isophorone	50
2-Nitrophenol	50
2,4-Dimethylphenol	50
Bis(2-chloroethoxy)methane	50
2,4-Dichlorophenol	50
1,2,4-Trichlorobenzene	50
Naphthalene	50
Hexachlorobutadiene	50
4-Chloro-3-methylphenol	50
Hexachlorocyclopentadiene	50
2,4,6-Trichlorophenol	50
2-Chloronaphthalene	50
Dimethyl phthalate	50
Acenaphthylene	50
Acenaphthene	50
2,4-Dinitrophenol	
4-Nitrophenol	50
2,4-Dinitrotoluene	050
2,6-Dinitrotoluene	
Diethylphthalate	50
4-Chlorophenyl phenyl ether	50
Fluorene	50
4,6-Dinitro-2-methylphenol	50
N-Nitrosodiphenylamine	50
4-Bromophenyl phenyl ether	50
Hexachlorobenzene	50
Pentachlorophenol	50
Phenanthrene	50
Anthracene	50
Di-n-butyl phthalate	50
Fluoranthene	50
Benzidine	50
Pyrene	50
Butyl benzyl phthalate	50
3,3'-Dichlorobenzidine	50
Benzo(a)anthracene	50

LCS Compounds	Spiking Level, ng/µL in extract[1]
Bis(2-ethylhexyl)phthalate	50
Chrysene	50
Di-n-octylphthalate	50
Benzo(b)fluoranthene	50
Benzo(k)fluoranthene	50
Benzo(a)pyrene	50
Indeno(1,2,3-cd)pyrene	50
Dibenz(a,h)anthracene	50
Benzo(g,h,i)perylene	50

Attachment QAPP-C5

Determination of Volatile Organics by GC/MS
Method 8260B, 625 and 524

SOP No.:	**CORP-MS-0002STL**
Revision No.:	1.3
Revision Date:	10/01/01
Page:	1 of 55
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STL ST. LOUIS STANDARD OPERATING PROCEDURE

TITLE: DETERMINATION OF VOLATILE ORGANICS BY GC/MS BASED ON METHOD 8260B, 624 AND 524.2

(SUPERSEDES: REVISION 1.2)

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Laboratory Director

1. SCOPE AND APPLICATION

1.1. This SOP is applicable to the determination of Volatile Organic Compounds in waters, wastewater, soils, sludges and other solid matrices. Standard analytes are listed in Tables 5 and 6.

1.2. This SOP is applicable to method 8260B. At client request, it may also be used for analysis following method 8240B. Appendices A, B and C present modifications to the procedures in the main SOP that are necessary for analysis of drinking water by method 524.2 and wastewater by method 624 and 8260A.

1.3. This method can be used to quantify most volatile organic compounds that have boiling points below 200°C and are insoluble or slightly soluble in water. Volatile water soluble compounds can be included in this analytical technique; however, for more soluble compounds, quantitation limits are approximately ten times higher because of poor purging efficiency.

1.4. The method is based upon a purge and trap, gas chromatograph/mass spectrometric (GC/MS) procedure. The approximate working range is 5 to 200 µg/L for 5 mL waters, 1 to 60 µg/L for 25 mL purge waters, 5 to 200 µg/kg for low-level soils, and 250 to 25,000 µg/kg for medium-level soils. Reporting limits are listed in Tables 1, 3 and A-1. Reporting limits will be proportionately higher for sample extracts that require dilution and for soil samples that require concentration adjustments to account for % moisture.

1.5. Method performance is monitored through the use of surrogate compounds, matrix spike/matrix spike duplicates, and laboratory control spike samples.

1.6. Method detection limits are maintained in the Information Management System (QuantIMS). Because of their dynamic nature, they are not specifically listed in this document, but can be retrieved at any time using TraQAr tools.

1.7. Quality control limits (accuracy and precision for spikes) are also maintained in QuantIMS, and are also dynamic. Therefore, they are not specifically listed in this document, but can be retrieved at any time using TraQAr tools.

1.8. Additional compounds may be amenable to this method. The minimum requirement for non-standard analytes is that the reporting limit be set at the lowest required concentration that can actually be detected by the instrument, and when an MDL study can not be conducted, the MDL be set equal to the reporting limit.

2. SUMMARY OF METHOD

2.1. Volatile compounds are introduced into the gas chromatograph by the purge and trap method. The components are separated via the chromatograph and detected using a mass spectrometer, which is used to provide both qualitative and quantitative information.

2.2. Aqueous samples are purged directly. Soils are preserved at 4°C for 5030 (see 11.7) or by extracting the volatile analytes into methanol. If low detection limits are required, soil samples may be preserved with sodium bisulfate and purged directly.

2.3. In the purge and trap process, an inert gas is bubbled through the solution at ambient temperature or at 40°C (40°C required for low level soils) and the volatile components are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbant column where the volatile components are trapped. After purging is completed, the sorbant column (trap) is heated and backflushed with inert gas to desorb the components onto a gas chromatographic column. The gas chromatographic column is then heated to elute the components that are detected with a mass spectrometer.

2.4. Qualitative identifications are confirmed by analyzing standards under the same conditions used for samples and comparing the resultant mass spectra and GC retention times. Each identified component is quantified by relating the MS response for an appropriate selected ion produced by that compound to the MS response for another ion produced by an internal standard.

3. DEFINITIONS

3.1. See policy QA-003 for definitions

3.2. Batch

The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. Using this method, each BFB analysis will normally start a new batch. Batches for medium level soils are defined at the sample preparation stage and may be analyzed on multiple instruments over multiple days, although reasonable effort should be made to keep the samples together.

3.2.1. The Quality Control batch must contain a matrix spike/spike duplicate (MS/MSD), a Laboratory Control Sample (LCS), and a method blank. In some cases, at client request, the MS/MSD may be replaced with a matrix spike and sample duplicate. Refer to the QC Program document (QA-003) for further details of the batch definition.

3.3. Method Blank

A method blank consisting of all reagents added to the samples must be analyzed with each batch of samples. The method blank is used to identify any background interference or contamination of the analytical system that may lead to the reporting of elevated concentration levels or false positive data.

3.4. Laboratory Control Sample (LCS)

Laboratory Control Samples are well characterized, laboratory generated samples used to monitor the laboratory's day-to-day performance of routine analytical methods. The LCS, spiked with a group of target compounds representative of the method analytes, is used to monitor the accuracy of the analytical process, independent of matrix effects. Ongoing monitoring of the LCS results provides evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision.

3.5. Surrogates

Surrogates are organic compounds which are similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which are not normally found in environmental samples. Each sample, blank, LCS, and MS/MSD is spiked with surrogate standards. Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits.

3.6. Matrix Spike/Matrix Spike Duplicates (MS/MSD)

A matrix spike is an environmental sample to which known concentrations of target analytes have been added. A matrix spike duplicate is a second aliquot of the same sample that is prepared and analyzed along with the sample and matrix spike. Matrix spikes and duplicates are used to evaluate accuracy and precision in the actual sample matrix.

3.7. Calibration Check Compound (CCC)

CCCs are a representative group of compounds that are used to evaluate initial calibrations and continuing calibrations. Relative percent difference for the initial calibration and % difference for the continuing calibration response factors are calculated and compared to the specified method criteria.

3.8. System Performance Check Compounds (SPCC)

SPCCs are compounds that are sensitive to system performance problems and are used to evaluate system performance and sensitivity. A response factor from the continuing calibration is calculated for the SPCC compounds and compared to the specified method criteria.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. The use of ultra high purity gases, pre-purged purified reagent water, and approved lots of purge and trap grade methanol will greatly reduce introduction of contaminants. In extreme cases the purging vessels may

be pre-purged to isolate the instrument from laboratory air contaminated by solvents used in other parts of the laboratory.

4.2. Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) into the sample through the septum seal during shipment and storage. A field blank prepared from reagent water and carried through the sampling and handling protocol can serve as a check on such contamination.

4.3. Matrix interferences may be caused by non-target contaminants that are coextracted from the sample. The extent of matrix interferences will vary considerably from source to source depending upon the nature and diversity of the site being sampled.

4.4. Cross-contamination can occur whenever high-level and low-level samples are analyzed sequentially or in the same purge position on an autosampler. Whenever an unusually concentrated sample is analyzed, it should be followed by one or more blanks to check for cross-contamination. The purge and trap system may require extensive bake-out and cleaning after a high-level sample.

4.5. Some samples may foam when purged due to surfactants present in the sample. When this kind of sample is encountered an antifoaming agent (e.g., J.T. Baker's Antifoam B silicone emulsion) can be used. A blank spiked with this agent must be analyzed with the sample because of the non-target interferences associated with the agent.

5. SAFETY

5.1. Procedures shall be carried out in a manner that protects the health and safety of all associates.

5.2. The Chemical Hygiene Plan (CHP) gives details about the specific health and safety practices that are to be followed in the laboratory area. Personnel must receive training in the CHP, including the written Hazard Communication plan, prior to working in the laboratory. Consult the CHP, the Health and Safety Policies and Procedures Manual, and available Material Safety Data Sheets (MSDS) prior to using the chemicals in the method.

5.3. Consult the Health and Safety Policies and Procedures Manual for information on Personal Protective Equipment. Eye protection that satisfies ANSI Z87.1 (as per the Chemical Hygiene Plan), laboratory coat and appropriate gloves must be worn while samples, standards, solvents and reagents are being handled. Disposable gloves that have become contaminated will be removed and discarded; other gloves will be cleaned immediately. VITON gloves may be worn when halogenated solvents are used for extractions or sample preparation. Nitrile gloves may be used when other solvents are handled.

5.4. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the MSDS files maintained in the laboratory. The following specific hazards are known:

5.4.1. Chemicals that have been classified as carcinogens, or potential carcinogens, under OSHA include: Acrylonitrile, benzene, carbon tetrachloride, chloroform, 1,2-dibromo-3-chloropropane, 1,4-dichlorobenzene, and vinyl chloride.

5.4.2. Chemicals known to be flammable are: Methanol.

5.5. Exposure to chemicals will be maintained **as low as reasonably achievable,** therefore, unless they are known to be non-hazardous, all samples will be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.6. The preparation of all standards and reagents and glassware cleaning procedures that involve solvents will be conducted in a fume hood with the sash closed as far as the operations will permit or by other means of mechanical ventilation.

5.7. All work must be stopped in the event of a known, or potential compromise to the health or safety of any associate. The situation must be reported **immediately** to a laboratory supervisor.

5.8. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices outlined in the Health and Safety Manual. These employees must have training on the hazardous waste disposal practices initially upon assignment of these tasks, followed by an annual refresher training.

6. EQUIPMENT AND SUPPLIES

6.1. Microsyringes: Hamilton or equivalent for standard and sample spiking.

6.2. Syringe: 5 or 25 mL glass with Luerlok tip, if applicable to the purging device.

6.3. Balance: Analytical, capable of accurately weighing 0.0001 g, and a top-loading balance capable of weighing 0.1 g

6.4. Glassware:

6.4.1. Vials: 20 and 40 mL with screw caps and Teflon liners.

6.4.2. Volumetric flasks: 10 mL and 100 mL, class A with ground-glass stoppers.

6.5. Spatula: Stainless steel.

6.6. Disposable pipettes: Pasteur.

6.7. pH paper: Wide range.

6.8. Gases:

6.8.1. Helium: Ultra high purity, gr. 5, 99.999%.

6.8.2. Nitrogen: Ultra high purity, from cylinders of gas generators, may be used as an alternative to helium for purge gas.

6.8.3. Compressed air: Used for instrument pneumatics.

6.8.4. Liquid nitrogen: Used for cryogenic cooling if necessary.

6.9. Purge and Trap Device: The purge and trap device consists of the sample purger, the trap, and the desorber.

6.9.1. Sample Purger: The recommended purging chamber is designed to accept 5 mL or 25ml samples with a water column at least 3 cm deep. The purge gas must pass through the water column as finely divided bubbles, each with a diameter of less than 3 mm at the origin. The purge gas must be introduced no more than 5 mm from the base of the water column. Alternative sample purge devices may be used provided equivalent performance is demonstrated. Low level soils are purged directly from a VOA vial.

6.9.2. Trap: A variety of traps may be used, depending on the target analytes required. For most purposes the Vocarb 3000 trap is suitable. Other traps, such as Vocarb 4000, or Tenax / Silica gel / Charcoal may be used if the Quality Control criteria are met.

6.9.3. Desorber: The desorber should be capable of rapidly heating the trap to 180°C. Many such devices are commercially available.

6.9.4. Sample Heater: A heater capable of maintaining the purge device at 40°C is necessary for low level soil analysis.

6.10. Gas Chromatograph/Mass Spectrometer System:

6.10.1. Gas Chromatograph: The gas chromatograph (GC) system must be capable of temperature programming.

6.10.2. Gas Chromatographic Columns: Capillary columns are used. Some typical columns are listed below:

6.10.2.1. Column 1: 105m x 0.53mm ID Rtx-502.2 with 3 µm film thickness.

6.10.2.2. Column 2: 60m x 0.25mm ID Rtx-VMS or Rtx-502.2 with 1.4 µm film thickness.

6.10.2.3. Mass Spectrometer: The mass spectrometer must be capable of scanning 35-300 AMU every two seconds or less, using 70 volts electron energy in the electron impact mode and capable of producing a mass spectrum that meets the required criteria when 50ng of 4-Bromofluorobenzene (BFB) are injected onto the gas chromatograph column inlet.

6.10.3. GC/MS interface: In general glass jet separators are used but any interface (including direct introduction to the mass spectrometer) that achieves all acceptance criteria may be used.

6.10.4. Data System: A computer system that allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that allows searching any GC/MS data file for ions of a specified mass and

plotting such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between for a specified time or scan-number limit. Also, for the non-target compounds, software must be available that allows for the comparison of sample spectra against reference library spectra. The most recent release of the NIST/EPA mass spectral library should be used as the reference library. The computer system must also be capable of backing up data for long-term off-line storage.

6.10.5. Cryogenic Cooling: Some columns require the use of liquid nitrogen to achieve the sub-ambient temperature required for the proper separation of the gases.

7. REAGENTS AND STANDARDS

7.1. Reagents

7.1.1. All reagent preparation is documented in the reagent logbook. All reagents are labeled with their unique ID, name (and concentration, if applicable) of the reagent, the date prepared and the expiration date.

7.1.2. Methanol: Purge and Trap Grade, High Purity

7.1.3. Reagent Water: High purity water that meets the requirements for a method blank when analyzed. (See section 9.2.4) Reagent water may be purchased as commercial distilled water and prepared by purging with an inert gas overnight. Other methods of preparing reagent water are acceptable.

7.2. Standards

7.2.1. All standards preparation, documentation and labeling must follow the requirements of STL-QA-0002.

7.2.2. Calibration Standard

7.2.2.1. Stock Solutions: Stock solutions may be purchased as certified solutions from commercial sources or prepared from pure standard materials as appropriate. These standards are prepared in methanol and stored in Teflon-sealed screw-cap bottles with minimal headspace at 0° to -20°C.

7.2.2.2. Working standards: A working solution containing the compounds of interest prepared from the stock solution(s) in methanol. These standards are stored in the freezer or as recommended by the manufacturer. Working standards are monitored by comparison to the initial calibration curve. If any of the calibration check compounds drift in response from the initial calibration by more than 20% then corrective action is necessary. This may include steps such as instrument maintenance, preparing a new calibration verification standard or tuning the instrument. If the corrective actions do not correct the problem then a new initial calibration must be performed.

7.2.2.3. Aqueous Calibration Standards are prepared in reagent water using the secondary dilution standards. These aqueous standards must be prepared daily.

7.2.2.4. If stock or secondary dilution standards are purchased in sealed ampoules they may be used up to the manufacturers expiration date.

7.2.2.5. Initial calibration verification (ICV) standards are similar to calibration standards, but are from a completely different source.

7.2.3. Internal Standards: Internal standards are added to all samples, standards, and blank analyses. Refer to Table 7 for internal standard components.

7.2.4. Surrogate Standards: Refer to Table 8 for surrogate standard components and spiking levels.

7.2.5. Laboratory Control Sample Spiking Solutions: Refer to Table 9 for LCS components and spiking levels.

7.2.6. Matrix Spiking Solutions: The matrix spike contains the same components as the LCS. Refer to Table 9.

7.2.7. Tuning Standard: A standard is made up that will deliver 50ng on column upon injection. A recommended concentration of 4-Bromofluorobenzene is 25ng/µL.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Holding times for all volatile analysis are 14 days from sample

8.2. Water samples are normally preserved at pH $\leq$ 2 with 1:1 hydrochloric acid. If residual chlorine is present, 2 drops of 10% sodium thiosulfate are added.

8.3. Solid samples are field preserved with sodium bisulfate solution for low level analysis, or with methanol for medium level analysis. Soil samples can also be taken using the EnCore™ sampler and preserved in the lab within 48 hours of sampling. At specific client request, unpreserved soil samples may be accepted.

8.4. There are several methods of sampling soil. The recommended method, which provides the minimum of field difficulties, is to take an EnCore sample. (The 5g or 25g sampler can be used, depending on client preference). Following shipment back to the lab the soil is preserved in methanol. This is the medium level procedure. If very low detection limits are needed (< 50 µg/kg for most analytes) then it will be necessary to use two additional 5 g EnCore samplers or to use field preservation.

8.5. Sample collection for medium level analysis using EnCore samplers.

8.5.1. Ship one 5 g (or 25 g) EnCore sampler per field sample position.

8.5.2. An additional bottle must be shipped for percent moisture determination.

8.5.3. When the samples are returned to the lab, extrude the (nominal) 5g (or 25g) sample into a tared VOA vial containing 5mL methanol (25mL methanol for the 25g sampler). Obtain the weight of the soil added to the vial and note on the label.

8.5.4. Add the correct amount of surrogate spiking mixture. (Add 50 µL of 1250 µg/mL solution for a nominal 25g sample, 10µL for a nominal 5g sample.)

8.5.5. Add the correct amount of matrix spiking solution to the matrix spike and matrix spike duplicate samples. (Add 50 µL of 1250µg/mL solution for a nominal 25g sample, 10µL for a nominal 5g sample.) The addition of spike introduces a slight error (0.4%), which can be neglected, into the calculations.

8.5.6. Prepare an LCS for each batch by adding the correct amount of matrix spiking solution to clean methanol. (50 µL of spike to 25mL methanol or 10 µL spike to 5 mL methanol).

8.5.7. Shake the samples for two minutes to distribute the methanol throughout the soil.

8.5.8. Allow to settle, then remove a portion of methanol to store in a clean Teflon capped vial at 4+/-2°C until analysis.

8.6. Sample collection for medium level analysis using field methanol preservation

8.6.1. Prepare a 2 oz sample container by adding 25 mL purge and trap grade methanol. (If a 5 g sample is to be used, add 5 mL methanol to a 2 oz container or VOA vial).

8.6.2. Seal the bottle and attach a label.

8.6.3. Weigh the bottle to the nearest 0.01g and note the weight on the label.

8.6.4. Ship with appropriate sampling instructions.

8.6.5. Each sample will require an additional bottle with no preservative for percent moisture determination.

8.6.6. At client request, the methanol addition and weighing may also be performed in the field.

8.6.7. When the samples are returned to the lab, obtain the weight of the soil added to the vial and note on the label.

8.6.8. Add the correct amount of surrogate spiking mixture. (Add 50 µL of 1250 µg/mL solution for a nominal 25 g sample, 10µL for a nominal 5 g sample.)

8.6.9. Add the correct amount of matrix spiking solution to the matrix spike and matrix spike duplicate samples. (Add 50 µL of 1250 µg/mL solution for a nominal 25 g sample, 10µL for a nominal 5 g

sample.) The addition of spike introduces a slight error, (0.2%) which can be neglected, into the calculations.

8.6.10. Prepare an LCS for each batch by adding the correct amount of matrix spiking solution to clean methanol. (50 µL of spike to 25 mL methanol or 10 µL spike to 5 mL methanol).

8.6.11. Shake the samples for two minutes to distribute the methanol throughout the soil.

8.6.12. Allow to settle, then remove a portion of methanol to store in a clean Teflon capped vial at 4+/-2°C until analysis.

8.7. Low level procedure

8.7.1. If low detection limits are required (typically < 50 µg/kg) sodium bisulfate preservation must be used. However, it is also necessary to take a sample for the medium level (field methanol preserved or using the EnCore sampler) procedure, in case the concentration of analytes in the soil is above the calibration range of the low level procedure.

8.7.2. A purge and trap autosampler capable of sampling from a sealed vial is required for analysis of samples collected using this method. (Tekmar Precept, Varian Archon, or O.I. 4552).

8.7.3. The soil sample is taken using a 5g EnCore sampling device and returned to the lab. It is recommended that two EnCore samplers be used for each field sample position, to allow for any reruns than may be necessary. A separate sample for % moisture determination is also necessary.

8.7.4. Prepare VOA vials by adding a magnetic stir bar, approximately 1 g of sodium bisulfate and 5 mL of reagent water.

8.7.5. Seal and label the vial. It is strongly recommended that the vial be labeled with an indelible marker rather than a paper label, since paper labels may cause the autosampler to bind and malfunction. The label absolutely must not cover the neck of the vial or the autosampler will malfunction.

8.7.6. Weigh the vial to the nearest 0.1g and note the weight on the label.

8.7.7. Extrude the soil sample from the EnCore sampler into the prepared VOA vial. Reweigh the vial to obtain the weight of soil and note on the label.

8.7.8. **Note:** Soils containing carbonates may effervesce when added to the sodium bisulfate solution. If this is the case at a specific site, add 5 mL of water instead, and freeze at <10°C until analysis.

8.7.9. Alternatively the sodium bisulfate preservation may be performed in the field. This is not recommended because of the many problems that can occur in the field setting. Ship at least two vials per sample. The field samplers must determine the weight of soil sampled. Each sample will require an additional bottle with no preservative for percent moisture determination, and an additional bottle preserved with methanol for the medium level procedure. Depending on the type of soil it may also be necessary to ship vials with no or extra preservative.

8.8. Unpreserved soils

8.8.1. At specific client request unpreserved soils packed into glass jars or brass tubes may be accepted and subsampled in the lab. This is the old procedure based on method 5030A. It is no longer included and is likely to generate results that are biased low, possibly by more than an order of magnitude.

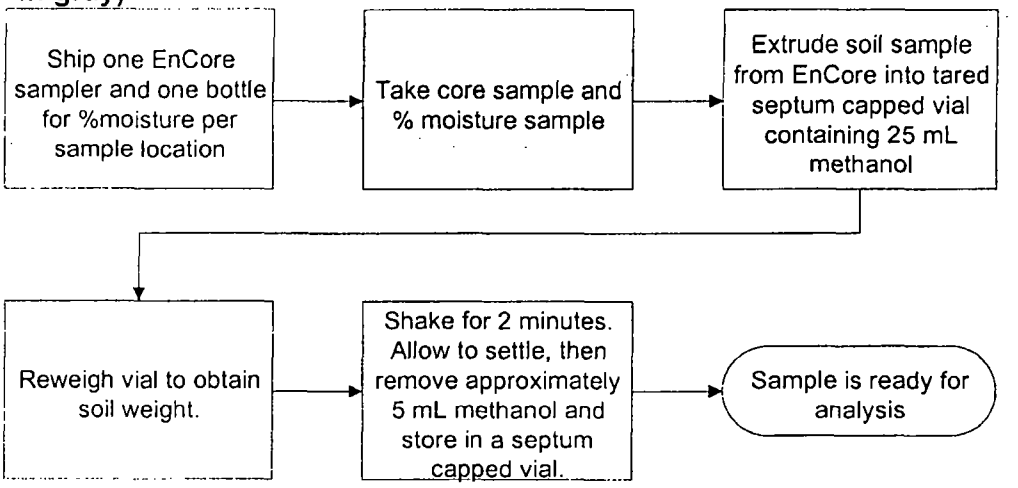
8.9. Aqueous samples are stored in glass containers with Teflon lined septa at 4°C +/- 2°C, with minimum headspace.

8.10. Medium level solid extracts are aliquoted into 2 - 5 mL glass vials with Teflon lined caps and stored at 4°C +/- 2°C. The extracts are stored with minimum headspace.

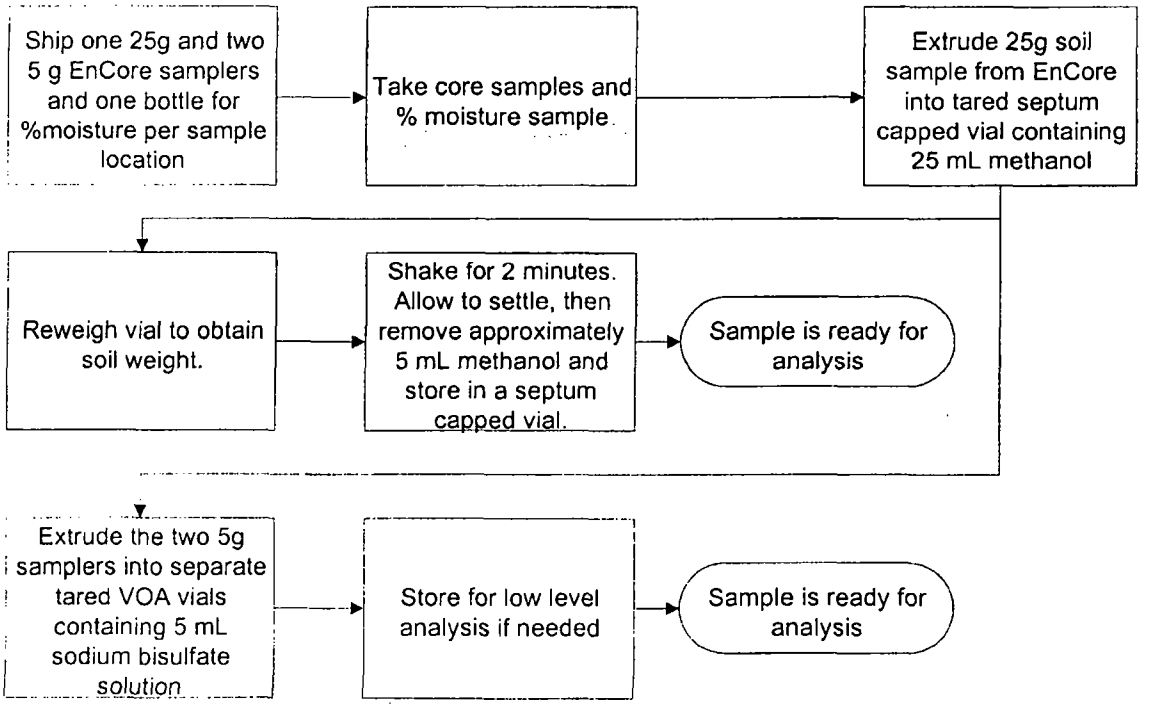
8.11. The maximum holding time is 14 days from sampling until the sample is analyzed. (Samples that are found to be unpreserved still have a 14-day holding time. However they should be analyzed as soon as possible. The lack of preservation should be addressed in the case narrative). Maximum holding time for the EnCore sampler (before the sample is added to methanol or sodium bisulfate) is 48 hours.

8.12. A holding blank is stored with the samples. This is analyzed and replaced if any of the trip blanks show any contamination. It is replaced in accordance with SOP SL-QA-0031, "VOA Holding Blank Analysis".

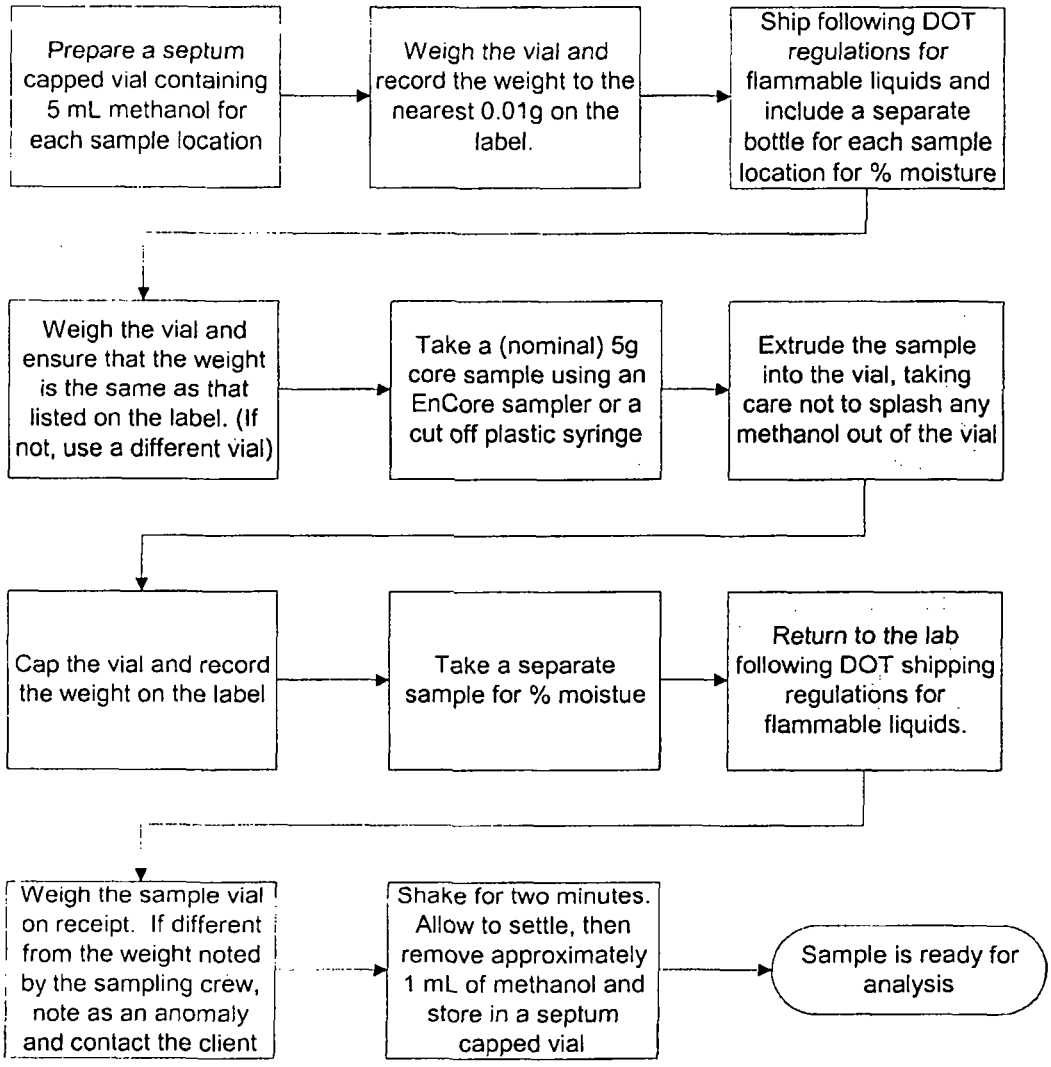
EnCore procedure when low level is not required (field steps in gray)



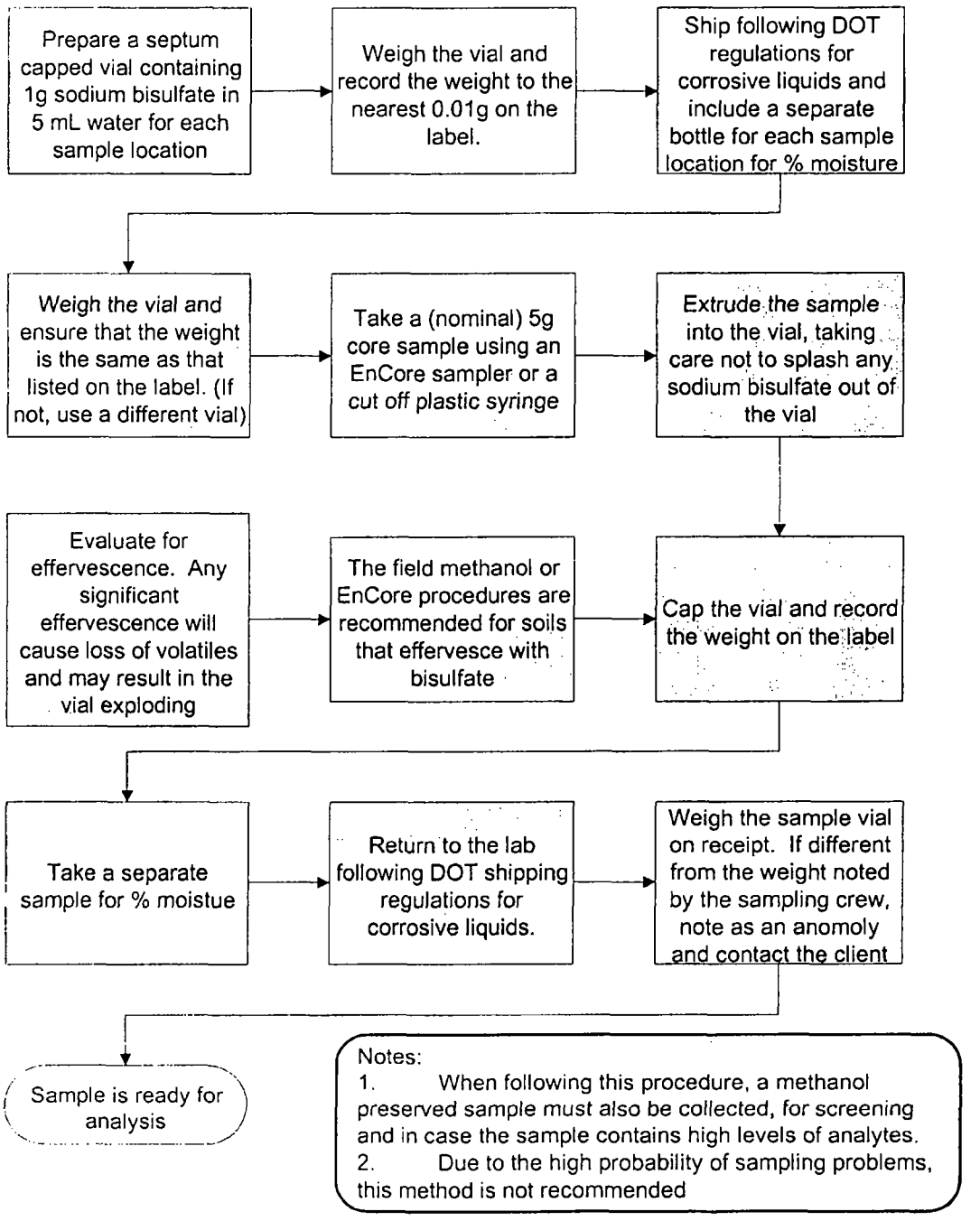
EnCore procedure when low level is required



Field methanol extraction procedure (field steps in gray)



Field bisulfate preservation procedure (field steps in gray)



9. QUALITY CONTROL

9.1. QC requirements

9.1.1. Each analytical batch may contain up to 20 environmental samples, a method blank, and a single Laboratory Control Sample (LCS) and an MS/MSD pair. In the event that there is not sufficient sample to analyze an MS/MSD, an LCS duplicate (LCSD) is prepared and analyzed.

9.1.1.1. Samples that have assigned QC limits different than the standard limits contained in QuantIMS QC code 01 must be batched separately, but can share the same QC samples.

9.1.1.2. Additional MS/MSDs do not count towards the 20 samples in an analytical batch.

9.1.1.3. A method blank must be included with each batch of samples.

9.1.1.4. At a minimum the LCS is spiked with a group of target compounds representative of the target analytes, and is used to monitor the accuracy of the analytical process, independent of matrix effects.

9.1.1.5. All LCS, MS/MSD, and surrogate results -- whether they pass criteria or not -- are uploaded into 's QuantIMS system for maintenance and periodic update of limits.

9.1.2. Instrument conditions must be the same for all standards, samples and QC samples.

9.1.3. All data will be reviewed by the analyst (1st review level) and then by a peer or supervisor (2nd level review).

9.2. Documentation

9.2.1. Initial Demonstration of Capability

9.2.1.1. For the standard analyte list, the initial demonstration described in Section 16 and method detection limit (MDL) studies must be acceptable before analysis of samples may begin. MDLs should be analyzed for low and medium soils and aqueous samples.

9.2.1.2. For non-standard analytes, a MDL study must be performed and calibration curve generated before analyzing any samples, unless lesser requirements are previously agreed to with the client. In any event, the minimum initial demonstration required is analysis of a standard at the reporting limit and a single point calibration.

9.2.2. Control Limits

In-house historical control limits must be determined for surrogates, matrix spikes, and laboratory control samples (LCS). These limits must be determined at least annually. The recovery limits are mean recovery +/- 3 standard deviations for surrogates, matrix spikes and LCS. Precision limits for matrix spikes / matrix spike duplicates are 0 to mean relative percent difference + 3 standard deviations.

9.2.2.1. All surrogate, LCS, and MS recoveries (except for dilutions) must be entered into QuantIMS (when available) or other database so that accurate historical control limits can be generated. For tests without a separate extraction, surrogates and matrix spikes will be reported for all dilutions.

9.2.2.2. Refer to the QC Program document (QA-003) for further details of control limits.

9.2.3. Surrogates

Every sample, blank, and QC sample is spiked with surrogates. Surrogate recoveries in samples, blanks, and QC samples must be assessed to ensure that recoveries are within established limits. The compounds included in the surrogate spiking solutions are listed in Table 8 and A-3. If any surrogates are outside limits, the following corrective actions must take place (except when surrogates are diluted out):

- Check all calculations for error.
- Ensure that instrument performance is acceptable.
- Recalculate the data and/or reanalyze if either of the above checks reveal a problem.
- Reprepare and reanalyze the sample or flag the data as "Estimated Concentration" if neither of the above resolves the problem.

The decision to reanalyze or flag the data should be made in consultation with the client. It is only necessary to reprepare/reanalyze a sample once to demonstrate that poor surrogate recovery is due to matrix effect, unless the analyst believes that the repeated out of control results are not due to matrix effect.

9.2.3.1. If the surrogates are out of control for the sample, matrix spike, and matrix spike duplicate, then matrix effect has been demonstrated for that sample and repreparation is not necessary. If the sample is out of control and the MS and/or MSD is in control, then reanalysis or flagging of the data is required.

9.2.3.2. Refer to the QC Program document (QA-003) for further details of the corrective actions.

9.2.4. Method Blanks

For each batch of samples, analyze a method blank. The method blank is analyzed after the calibration standards, normally before any samples. For low-level volatiles, the method blank consists of reagent water. The blank can be matrix matched at the client's request. For medium-level volatiles, the method blank consists of 5.0 or 25.0 mL of methanol depending on the sample weights in the batch. Surrogates are added and the method blank is carried through the entire analytical procedure. The method blank must not contain any analyte of interest at or above the reporting limit (except common laboratory contaminants, see below) or at or above 5% of the measured concentration of that analyte in the associated samples, whichever is higher.

- If the analyte is a common laboratory contaminant (methylene chloride, acetone, 2-butanone) the data may be reported with qualifiers if the concentration of the analyte is less than five times the reporting limit. Such action must be taken in consultation with the client.
- Reanalysis of samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples.
- If there is no target analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. Such action should be done in consultation with the client.

9.2.4.1. The method blank must have acceptable surrogate recoveries. If surrogate recoveries are not acceptable, the data must be evaluated to determine if the method blank has served the purpose of demonstrating that the analysis is free of contamination. If surrogate recoveries are low and there are reportable analytes in the associated samples reextraction of the blank and affected samples will normally be required. Consultation with the client should take place.

9.2.4.2. If reanalysis of the batch is not possible due to limited sample volume or other constraints, the method blank is reported, all associated samples are flagged with a "B", and appropriate comments may be made in a narrative to provide further documentation.

9.2.4.3. Refer to the QC Program document (QA-003) for further details of the corrective actions.

9.2.5. Laboratory Control Samples (LCS)

For each batch of samples, analyze a LCS. The LCS is analyzed after the calibration standard, and normally before any samples. The LCS contains a representative subset of the analytes of interest (See Table 9), and must contain the same analytes as the matrix spike. If any analyte or surrogate is outside established control limits, the system is out of control and corrective action must occur. Corrective action will normally be repreparation and reanalysis of the batch.

- If the batch is not re-extracted and reanalyzed, the reasons for accepting the batch must be clearly presented in the case narrative and the client notified.
- If re-extraction and reanalysis of the batch is not possible due to limited sample volume or other constraints, the LCS is reported, all associated samples are flagged, and appropriate comments are made in a narrative to provide further documentation.

9.2.5.1 Refer to the QC Program document (QA-003) for further details of the corrective action.

9.2.5.2 If full analyte spike lists are used at client request, it will be necessary to allow a percentage of the components to be outside control limits as this would be expected statistically. These requirements should be negotiated with the client.

9.2.6 Matrix Spikes

For each QC batch, analyze a matrix spike and matrix spike duplicate. Spiking compounds and levels are given in Table 9. Compare the percent recovery and relative percent difference (RPD) to that in the laboratory specific historically generated limits.

- If any individual recovery or RPD falls outside the acceptable range, corrective action must occur. The initial corrective action will be to check the recovery of that analyte in the Laboratory Control Sample (LCS). Generally, if the recovery of the analyte in the LCS is within limits, then the laboratory operation is in control and analysis may proceed. The reasons for accepting the batch must be documented.
- If the recovery for any component is outside QC limits for both the matrix spike/ spike duplicate and the LCS, the laboratory is out of control and corrective action must be taken. Corrective action will normally include reanalysis of the batch.
- If a MS/MSD is not possible due to limited sample, then a LCS duplicate should be analyzed. RPD of the LCS and LCSD are compared to the matrix spike limits.
- The matrix spike/duplicate should be analyzed at the same dilution as the unspiked sample. If the matrix spike compounds will be diluted out perhaps another sample could be chosen or client consultation should occur.

9.2.7 Nonconformance and Corrective Action

Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the facility QA Manager.

9.2.8 Quality Assurance Summaries

Certain clients may require specific project or program QC that may supersede these method requirements. These requirements should be addressed in the Client Requirement Checklist.

9.2.9 QC Program

Further details of QC and corrective action guidelines are presented in the QC Program document (QA-003). Refer to this document if in doubt regarding corrective actions.

9.3. Procedural Variations

9.3.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of the analyst to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

9.3.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

9.4. Nonconformance and Corrective Action

Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the facility QA Manager.

9.5. QC Program

Further details of QC and corrective action guidelines are presented in the QC Program policy document (QA-003).

10. CALIBRATION AND STANDARDIZATION

10.1. Summary

10.1.1. Prior to the analysis of samples and blanks, each GC/MS system must be tuned and calibrated. Hardware tuning is checked through the analysis of the 4-Bromofluorobenzene (BFB) to establish

that a given GC/MS system meets the standard mass spectral abundance criteria. The GC/MS system must be calibrated initially at a minimum of five concentrations (analyzed under the same BFB tune), to determine the linearity of the response utilizing target calibration standards. Once the system has been calibrated, the calibration must be verified each twelve hour time period for each GC/MS system. The use of separate calibrations is required for water and low soil matrices.

10.2. Recommended Instrument Conditions

10.2.1. General

Electron Energy:	70 volts (nominal)
Mass Range:	35–300 AMU
Scan Time:	to give at least 5 scans/peak, but not to exceed 2 second/scan
Injector Temperature:	200–250°C
Source Temperature:	According to manufacturer's specifications
Transfer Line	Temperature: 250–300°C
Purge Flow:	40 mL/minute
Carrier Gas	Flow: 15 mL/minute
Make-up Gas Flow:	25–30 mL/minute

10.2.2. Gas chromatograph suggested temperature program

10.2.2.1. BFB Analysis

Isothermal:	170°C

10.2.2.2. Sample Analysis

Initial Temperature:	40°C
Initial Hold Time:	4 minutes
Temperature Program:	8°C/minute
Final Temperature:	184°C
Second Temperature	Program: 40°C/minute
Final Temperature:	240°C
Final Hold Time: 2.6 minutes	

10.3. Instrument Tuning

10.3.1. Each GC/MS system must be hardware-tuned to meet the abundance criteria listed in Table 10 for a maximum of a 50 ng injection or purging of BFB. Analysis must not begin until these criteria are met. These criteria must be met for each twelve-hour time period. The twelve-hour time period begins at the moment of injection of BFB.

10.4. Initial Calibration

10.4.1. A series of five initial calibration standards is prepared and analyzed for the target compounds and each surrogate compound. Typical calibration levels for a 5 mL purge are 5, 20, 50, 100, and 200 µg/L. Certain analytes are prepared at higher concentrations due to poor purge performance. Typical calibration levels for a 25 mL purge are 1, 5, 10, 30, and 60 µg/L. Again, some analytes are prepared at higher levels. Tables 2, 4, and A-4 list the calibration levels for each analyte. Other calibration levels and purge volumes may be used depending on the capabilities of the specific instrument. However, the same purge volume must be used for calibration and sample analysis, and the low level standard must be at or below the reporting limit.

10.4.2. It may be necessary to analyze more than one set of calibration standards to encompass all of the analytes required for same tests. For example, the Appendix IX list requires the Primary standard (Table 5) and the Appendix IX standard (Table 6). If acceptable analytical performance can be obtained the primary and appendix IX standards may be analyzed together.

10.4.3. Internal standard calibration is used. The internal standards are listed in Tables 7 and A-2. Target compounds should reference the nearest internal standard. Each calibration standard is analyzed and the response factor (RF) for each compound is calculated using the area response of the characteristic ions against the concentration for each compound and internal standard. See equation 1, Section 12, for calculation of response factor.

10.4.4. The % RSD of the calibration check compounds (CCC) must be less than 30%. Refer to Table 12 for the CCCs.

10.4.4.1. If none of the CCCs are required analytes, project specific calibration specifications must be agreed with the client.

10.4.5. The average RF must be calculated for each compound. A system performance check is made prior to using the calibration curve. The five system performance check compounds (SPCC) are checked for a minimum average response factor. Refer to Table 11 for the SPCC compounds and required minimum response factors.

10.4.5.1. If the average of all %RSDs in the calibration is $\leq$ 15%, then all analytes may use average response factor for calibration.

10.4.5.2. If the software in use is capable of routinely reporting curve coefficients for data validation purposes, and the necessary calibration reports can be generated, then the analyst should evaluate analytes with %RSD > 15% for calibration on a curve. If it appears that substantially better accuracy would be obtained using quantitation from a curve then the appropriate curve should be used for quantitation. If Relative Standard Error (RSE) is used to evaluate the curve it must be better than 15%. Otherwise the correlation coefficient (coefficient of determination for non-linear curves) must be $\geq$ 0.990.

10.4.5.3. If the average of all %RSDs in the calibration is > 15% then calibration on a curve must be used for all analytes with %RSD > 15%. The analyst should consider instrument maintenance to improve the linearity of response. If Relative Standard Error (RSE) is used to evaluate the curve it must be better than 15%. Otherwise the correlation coefficient, r (coefficient of determination, r^2 for non-linear curves) must be $\geq$ 0.990.

10.4.6. Weighting of Data Points

In a linear or quadratic calibration fit, the points at the lower end of the calibration curve have less weight in determining the curve generated than points at the high concentration end of the curve. However, in environmental analysis, accuracy at the low end of the curve is very important. For this reason it is preferable to increase the weighting of the lower concentration points. $1/Concentration^2$ weighting (often called $1/X^2$ weighting) will improve accuracy at the low end of the curve and should be used if the data system has this capability.

10.4.7. If time remains in the 12-hour period initiated by the BFB injection before the initial calibration, samples may be analyzed. Otherwise, proceed to continuing calibration.

10.4.8. A separate five-point calibration must be prepared for analysis of low level soils. Low level soil analysis requires the use of a closed vial autosampler such as the Varian Archon, O.I. 4552 or Tekmar Precept. Each standard is prepared by spiking the methanolic standard solution through the septum of a VOA vial containing 5 mL of water and 1 g sodium bisulfate. The standards are heated to 40°C for purging. All low-level soil samples, standards, and blanks must also be heated to 40°C for purging. Medium soil extracts should be analyzed using the water (unheated) calibration curve.

10.4.9. Non-standard analytes are sometimes requested. For these analytes, it is acceptable to analyze a single standard at the reporting limit with each continuing calibration rather than a five point initial calibration. If the analyte is detected in any of the samples, a five point initial calibration must be generated and the sample(s) reanalyzed for quantitation. However, if the analyte is not detected, the non-detect may be reported and no further action is necessary.

10.4.10. If the calibration curve does not meet method requirements,

10.4.10.1. Evaluate whether the problem is related to the analytical range. The low standard or the high standard having a response that is out of line with the others typically expresses this. In such situations, the analyst should determine if the analytical range should be decreased by deleting the high or low standard. If the low standard is deleted, adjust the reporting limit. If the high standard is deleted, dilute samples appropriately. If the high standard shows saturation of the response, the calibration range must be adjusted.

10.4.10.2. Evaluate whether the problem was related to a mis-injection of one of the standards. This must be confirmed by reanalyzing the suspect standard. If the reanalyzed standard is in line with the others, the mis-injection of the original standard is confirmed and must be replaced. The minimum number of calibration points must be five.

10.4.10.3. The relationship between the standard mass and the response of each compound in the calibration standards can be fitted to a linear regression curve. However, this should not be done for compounds that normally pass average RRF curve requirements (see 10.4.5 for considerations). In all cases, the coefficient of determination (R^2) for all regression curves must be equal to or greater than 0.990.

10.5. Initial calibration verification

10.5.1. A second source ICV will be analyzed following each IC. Since the regulatory agencies have not provided guidance on Second Source Standards checking, STL has set the following recovery criteria based on experience to date on several methods:

- General criteria ± 35%
- Warning limits between ± 35 - 55%

Note – It is desirable to have all non-control analytes within control limits, but it is expected that a small proportion will not be in control at any given time. By nature of their chemical properties, some analytes will inherently be found in the warning limits (WL) and these analytes will be reviewed on a case by case basis. Based upon the professional judgement of the spectroscopist and QA Manager, a decision will be achieved that gauges the validity of the calibration and preparation of the standards.

10.6. Continuing calibration verification.

Note - A CCV standard need not be analyzed in tune periods when an initial calibration curve was analyzed.

10.7. Continuing Calibration: The initial calibration must be verified every twelve hours.

10.7.1. Continuing calibration begins with analysis of BFB as described in Section 10.3. If the system tune is acceptable, the continuing calibration standard(s) are analyzed. The level 3 calibration standard is used as the continuing calibration.

10.7.2. The RF data from the standards are compared with the average RF from the initial five-point calibration to determine the percent difference of the CCC compounds. The calculation is given in equation 4, Section 12.3.4.

10.7.3. The % drift of the CCCs must be $\leq$ 20% for the continuing calibration to be valid. The SPCCs are also monitored. The SPCCs must meet the criteria described in Table 11. In addition, the % difference of all analytes must be $\leq$ 50% with allowance for up to six target analytes to have % difference > 50%.

10.7.3.1. If none of the CCCs are required analytes, project specific calibration specifications must be agreed with the client.

10.7.3.2. Cyclohexanone, one of the components of the Appendix IX standard, is unstable in the calibration solution, forming 1,1-dimethoxycyclohexane. No calibration criteria are applied to cyclohexanone and quantitation is tentative. Cyclohexanone is included on the Universal Treatment Standard and FO-39 regulatory lists (but not on Appendix IX).

10.7.4. If the CCCs and or the SPCCs do not meet the criteria in Sections 10.4.4 and 10.4.5, the system must be evaluated and corrective action must be taken. The BFB tune and continuing calibration must be

acceptable before analysis begins. Extensive corrective action such as a different type of column will require a new initial calibration.

10.7.5. Once the above criteria have been met, sample analysis may begin. **Initial calibration average RFs (or the calibration curve) will be used for sample quantitation, not the continuing calibration RFs.** Analysis may proceed until 12 hours from the injection of the BFB have passed. (A sample *desorbed* less than or equal to 12 hours after the BFB is acceptable.)

10.7.6. The CCV must be within the %D criteria specified at the beginning of the appropriate method-specific section.

Note – When an analyte is not detected in associated samples, strict calibration criteria can be relaxed. This is expressed as maximum %D in the appropriate method-specific sections.

10.7.7. In some methods, additional continuing calibration requirements may exist (*e.g.*, internal standards, minimum RRF requirement for certain analytes). Consult the method-specific section for applicability.

11. PROCEDURE

11.1. Preliminary Evaluation

11.1.1. Where possible, samples are screened by headspace or GC/MS off-tune analysis to determine the correct aliquot for analysis. Alternatively, an appropriate aliquot can be determined from sample histories.

11.1.2. Dilutions should be done just prior to the GC/MS analysis of the sample. Dilutions are made in volumetric flasks or in a Luerlok syringe. Calculate the volume of reagent water required for the dilution. Fill the syringe with reagent water, compress the water to vent any residual air and adjust the water volume to the desired amount. Adjust the plunger to the mark and inject the proper aliquot of sample into the syringe. If the dilution required would use less than 5 µL of sample then serial dilutions must be made in volumetric flasks.

11.1.3. The diluted concentration is to be estimated to be in the upper half of the calibration range.

11.2. Sample Analysis Procedure

11.2.1. All analysis conditions for samples must be the same as for the continuing calibration standards (including purge time and flow, desorb time and temperature, column temperatures, multiplier setting etc.).

11.2.2. All samples must be analyzed as part of a batch. The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. The batch also must contain a MS/MSD, a LCS, and a method blank.

11.2.2.1. If there is insufficient time in the 12-hour tune period to analyze 20 samples, the batch may be continued into the next tune period. However, if any re-tuning of the instrument is necessary, or if a period of greater than 24 hours from the preceding BFB tune has passed, a new batch must be started. For medium level soils the batch is defined at the sample preparation stage.

11.2.2.2. Laboratory generated QC samples (Blank, LCS, MS/MSD) do not count towards the maximum 20 samples in a batch. Field QC samples are included in the batch count.

11.2.2.3. It is not necessary to reanalyze batch QC with reanalyses of samples. However, any reruns must be as part of a valid batch.

11.3. Water Samples

11.3.1. All samples and standard solutions must be at ambient temperature before analysis.

11.3.2. Fill a syringe with the sample. If a dilution is necessary it may be made in the syringe if the sample aliquot is $\geq$ 5 µL. Check and document the pH of the remaining sample.

11.3.3. Add 250 ng of each internal and surrogate standard (10 µL of a 25 µg/mL solution, refer to Tables 7, 8, 15 and 16). The internal standards and the surrogate standards may be mixed and added as one spiking solution (this results in a 50 µg/L solution for a 5 mL sample, and a 10 µg/L solution for a 25 mL sample). Inject the sample into the purging chamber.

11.3.3.1. For TCLP samples use 0.5 mL of TCLP leachate with 4.5 mL reagent water and spike with 10 µL of the 25 µg/mL TCLP spiking solution. (Note that TCLP reporting limits will be 10 times higher than the corresponding aqueous limits).

11.3.3.2. Typically the sample is purged for ten minutes with the trap below 35°C.

11.3.4. After purging is complete, desorb the sample, start the GC temperature program, and begin data acquisition. After desorption, bake the trap for 5-10 minutes to condition it for the next analysis. When the trap is cool, it is ready for the next sample.

11.3.5. Desorb and bake time and temperature are optimized for the type of trap in use. The same conditions must be used for samples and standards.

11.4. Methanol Extract Soils

11.4.1. Rinse a gas-tight syringe with organic free water. Fill the syringe with the same volume of organic free water as used in the calibrations. Add no more than 2% (v/v) (100 µL for a 5 mL purge) methanolic extract (from Section 8.5 or 8.6) to the syringe. Add internal standard (if used). Load the sample onto the purge and trap device and analyze as for aqueous samples. If less than 5µL of methanolic extract is to be added to the water, dilute the methanolic extract such that a volume greater than 5µL will be added to the water in the syringe.

11.5. Liquid wastes that are soluble in methanol and insoluble in water.

11.5.1. Pipette 2 mL of the sample into a tared vial. Use a top-loading balance. Record the weight to the nearest 0.1 gram.

11.5.2. Quickly add 7 mL of methanol, then add 1 mL of surrogate spiking solution to bring the final volume to 10 mL. Cap the vial and shake for 2 minutes to mix thoroughly. For a MS/MSD or LCS, 6 mL of methanol, 1 mL of surrogate solution, and 1 mL of matrix spike solution is used.

11.5.3. Rinse a gas-tight syringe with organic free water. Fill the syringe with the same volume of organic free water as used in the calibrations. Add no more than 2% (v/v) (100 µL for a 5 mL purge) methanolic extract (from Section 8.5 or 8.6) to the syringe. Add internal standard (if used). Load the sample onto the purge and trap device and analyze as for aqueous samples. If less than 5µL of methanolic extract is to be added to the water, dilute the methanolic extract such that a volume greater than 5µL will be added to the water in the syringe.

11.6. Aqueous and Low level Soil Sample Analysis (Purge and Trap units that sample directly from the VOA vial)

11.6.1. Units that sample from the VOA vial should be equipped with a module that automatically adds surrogate and internal standard solution to the sample prior to purging the sample.

11.6.2. If the autosampler uses automatic IS/SS injection, no further preparation of the VOA vial is needed. *Otherwise the internal and surrogate standards must be added to the vial. Note:* Aqueous samples with high amounts of sediment present in the vial may not be suitable for analysis on this instrumentation, or they may need to analyzed as soils.

11.6.3. Soil samples must be quantitated against a curve prepared with standards containing about the same amount of sodium bisulfate as the samples (1 g in 5 mL).

11.6.4. Sample remaining in the vial after sampling with one of these mechanisms is no longer valid for further analysis. A fresh VOA vial must be used for further sample analysis.

11.6.5. For aqueous samples, check the pH of the sample remaining in the VOA vial after the sample aliquot has been taken.

11.6.6. Low-Level Solids Analysis using discrete autosamplers

Note: This technique may seriously underestimate analyte concentration and must not be used except at specific client request for the purpose of comparability with previous data. It is no longer part of SW-846.

This method is based on purging a heated soil/sediment sample mixed with reagent water containing the surrogates and internal standards. Analyze all reagent blanks and standards under the same conditions as the samples (e.g., heated). The calibration curve is also heated during analysis. Purge temperature is 40°C.

11.6.7. *Do not discard any supernatant liquids. Mix the contents of the container with a narrow metal spatula.*

11.6.8. *Weigh out 5 g (or other appropriate aliquot) of sample into a disposable culture tube or other purge vessel. Record the weight to the nearest 0.1 g. If method sensitivity is demonstrated, a smaller aliquot may be used. Do not use aliquots less than 1.0 g unless client notified. If the sample is contaminated with analytes such that a purge amount less than 1.0 g is appropriate, use the medium level method. For the medium level method, add 5 g soil to 5 mL methanol containing the surrogates, mix for two minutes, allow to settle then remove a portion of the methanol and store in a clean Teflon capped vial at 4 °C until analysis. Analyze as described in section 11.5.*

11.6.9. *Connect the purge vessel to the purge and trap device.*

11.6.10. *Rinse a 5 mL gas-tight syringe with organic free water, and fill. Compress to 5 mL. Add surrogate/internal standard (and matrix spike solutions if required.). Add directly to the sample from 11.5.2.*

11.6.11. *The above steps should be performed rapidly and without interruption to avoid loss of volatile organics.*

11.6.12. *Add the heater jacket or other heating device and start the purge and trap unit.*

11.6.13. *Soil samples that have low IS recovery when analyzed (<50%) should be reanalyzed once to confirm matrix effect.*

11.7. Initial review and corrective actions

11.7.1. If the retention time for any internal standard in the continuing calibration changes by more than 0.5 minutes from the mid-level initial calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Reanalysis of samples analyzed while the system was malfunctioning is required.

11.7.2. If the internal standard response in the continuing calibration is more than 200% or less than 50% of the response in the mid-level of the initial calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Reanalysis of samples analyzed while the system was malfunctioning is required.

11.7.2.1. Any samples that do not meet the internal standard criteria for the continuing calibration must be evaluated for validity. If the change in sensitivity is a matrix effect confined to an individual sample reanalysis is not necessary. If the change in sensitivity is due to instrumental problems all affected samples must be reanalyzed after the problem is corrected.

11.7.3. The surrogate standard recoveries are evaluated to ensure that they are within limits. Corrective action for surrogates out of control will normally be to reanalyze the affected samples. However, if the surrogate standard response is out high and there are no target analytes or tentatively identified compounds, reanalysis may not be necessary. Out of control surrogate standard response may be a matrix effect. It is only necessary to reanalyze a sample once to demonstrate matrix effect, but reanalysis at a dilution should be considered.

11.8. Dilutions

If the response for any compound exceeds the working range of the GC/MS system, a dilution of the extract is prepared and analyzed. An appropriate dilution should be in the upper half of the calibration range. Samples may be screened to determine the appropriate dilution for the initial run. If the initial diluted run has no hits or

hits below 20% of the calibration range and the matrix allows for analysis at a lesser dilution, then the sample must be reanalyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

11.8.1. Guidance for Dilutions Due to Matrix

If the sample is initially run at a dilution and the baseline rise is less than half the height of the internal standards, or if individual non target peaks are less than twice the height of the internal standards, then the sample should be reanalyzed at a more concentrated dilution. This requirement is approximate and subject to analyst judgement.

11.8.2. Reporting Dilutions

The most concentrated dilution with no target compounds above the calibration range will be reported. Other dilutions will only be reported at client request.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Qualitative identification

An analyte is identified by retention time and by comparison of the sample mass spectrum with the mass spectrum of a standard of the suspected compound (standard reference spectrum). Mass spectra for standard reference may be obtained on the user's GC/MS by analysis of the calibration standards or from the NIST Library. Two criteria must be satisfied to verify identification: (1) elution of sample component at the same GC retention time as the standard component; and (2) correspondence of the sample component and the standard component characteristic ions. (Note: Care must be taken to ensure that spectral distortion due to co-elution is evaluated.)

- The sample component retention time must compare to within ± 0.2 min. of the retention time of the standard component. For reference, the standard must be run within the same twelve hours as the sample.
- All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) should be present in the sample spectrum.
- The relative intensities of ions should agree to within ±30% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 20 and 80 percent.)

12.1.1. If a compound cannot be verified by all the above criteria, but in the technical judgment of the analyst, the identification is correct, then the analyst shall report that identification and proceed with quantitation.

12.2. Tentatively Identified Compounds (TICs)

12.2.1. If the client requests components not associated with the calibration standards, a search of the NIST library may be made for the purpose of tentative identification. Guidelines are:

12.2.1.1. Relative intensities of major ions in the reference spectrum (ions > 10% of the most abundant ion) should be present in the sample spectrum.

12.2.1.2. The relative intensities of the major ions should agree to within 20%. (Example: If an ion shows an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30% and 70%).

12.2.1.3. Molecular ions present in the reference spectrum should be present in the sample spectrum.

12.2.1.4. Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.

12.2.1.5. Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the spectrum because of background contamination or coeluting peaks. (Data system reduction programs can sometimes create these discrepancies.)

12.2.1.6. Computer-generated library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual inspection of the sample with the nearest library searches should the analyst assign a tentative identification.

12.3. Calculations.

12.3.1. Response factor (RF):

Equation 1

$$RF = \frac{A_x C_{is}}{A_{is} C_x}$$

Where:
A_x = Area of the characteristic ion for the compound to be measured
A_{is} = Area of the characteristic ion for the specific internal standard
C_{is} = Concentration of the specific internal standard, ng
C_x = Concentration of the compound being measured, ng

12.3.2. Standard deviation (SD):

Equation 2

$$SD = \sqrt{\sum_{i=1}^{N} \frac{(Xi - X)^2}{N - 1}}$$

X_i = Value of X at i through N
N = Number of points
X = Average value of X_i

12.3.3. Percent relative standard deviation (%RSD):

Equation 3

$$\%RSD = \frac{\text{Standard Deviation}}{\overline{RFi}} \times 100$$

$\overline{RFi}$ = **Mean of RF values in the curve**

12.3.4. Percent drift between the initial calibration and the continuing calibration:

Equation 4

$$\%Diff = \frac{RF_v - \overline{RF}}{\overline{RF}} \times 100$$

Where

RF_v = Response factor from continuing calibration standard

$\overline{RF}$ = Mean response factor from initial calibration

12.3.5. Target compound and surrogate concentrations:

Concentrations in the sample may be determined from linear or second order (quadratic) curve fitted to the initial calibration points, or from the average response factor of the initial calibration points. Average response factor may only be used when the % RSD of the response factors in the initial calibration is ≤ 15%.

12.3.5.1. Calculation of concentration using Average Response Factors

Equation 5

$$\textbf{Concentration } \mu g / L = \frac{x}{RF}$$

12.3.5.2. Calculation of concentration using Linear fit

Equation 6

$$\textbf{Concentration } \mu g / L = A + Bx$$

12.3.5.3. Calculation of concentration using Quadratic fit

Equation 7

$$\textbf{Concentration } \mu g / L = A + Bx + Cx^2$$

x is defined in equations 8, 9 and 10

A is a constant defined by the intercept

B is the slope of the curve

C is the curvature

12.3.5.4. Calculation of x for Water and water-miscible waste:

Equation 8

$$x = \frac{(A_x)(I_s)(D_f)}{(A_{is})(V_o)}$$

Where:

A_x = Area of characteristic ion for the compound being measured (secondary ion quantitation is allowed only when there are sample interferences with the primary ion)

A_{is} = Area of the characteristic ion for the internal standard

I_s = Amount of internal standard added in ng

$$\text{Dilution Factor} = D_f = \frac{\text{Total volume purged (mL)}}{\text{Volume of original sample used (mL)}}$$

V_o = Volume of water purged, mL

12.3.5.5. Calculation of x for Medium level soils:

Equation 9

$$x = \frac{(A_x)(I_s)(V_t)(1000)(D_f)}{(A_{is})(V_a)(W_s)(D)}$$

Where:
A_x, I_s, D_f, A_{is}, same as for water.
V_t = Volume of total extract, mL (Typically 25 mL)
V_a = Volume of extract added for purging, µL
W_s = Weight of sample extracted, g

$$D = \frac{100 - \%\text{moisture}}{100}$$

12.3.5.6. Calculation of x for Low level soils:

Equation 10

$$x = \frac{(A_x)(I_s)}{(A_{is})(W_s)(D)}$$

Where:
A_x, I_s, A_{is}, same as for water.
D is as for medium level soils
W_s = Weight of sample added to the purge vessel, g

12.3.5.7. Calculation of TICs: The calculation of TICs (tentatively identified compounds) is identical to the above calculations with the following exceptions:

A_x = Area in the total ion chromatogram for the compound being measured
A_{is} = Area of the total ion chromatogram for the nearest internal standard without interference
RF = 1
In other words, the concentration is equal to x as defined in equations 8, 9 and 10.

12.3.6. MS/MSD Recovery

Equation 11

$$\textbf{Matrix Spike Recovery, \%} = \frac{SSR - SR}{SA} \times 100$$

SSR = Spike sample result

SR = Sample result

SA = Spike added

12.3.7. Relative % Difference calculation for the MS/MSD

Equation 12

$$\mathbf{RPD} = \frac{|\mathbf{MSR} - \mathbf{MSDR}|}{\frac{1}{2}(\mathbf{MSR} + \mathbf{MSDR})} \times 100$$

Where:
RPD = Relative percent difference
MSR = Matrix spike result
MSDR = Matrix spike duplicate result

13. DATA ASSESSMENT AND ACCEPTANCE CRITERIA

The following represent data assessment for samples and acceptance criteria for QC measures. Corrective actions for any failure in QC measures are shown in Section 14.

13.1. QC sample acceptance criteria

13.1.1. Method Blank.

13.1.1.1. No target analytes may be present in the method blank above the reporting limit with the exception of the common laboratory solvents, methylene chloride and acetone.

13.1.1.2. The method blank must have acceptable surrogate recoverics.

13.1.2. Laboratory Control Sample (LCS).

13.1.2.1. All control analytes must be within established control limits for accuracy (%Recovery) and precision (RPD). Exceptions are allowed only with QA and project management approval.

13.1.2.2. It is desirable to have all non-control analytes within control limits, but it is expected that a small proportion will not be in control.

13.1.2.3. The LCS must have acceptable surrogate recoveries.

13.1.3. Matrix Spike/Matrix Spike Duplicate (MS/MSD).

13.1.3.1. All analytes should be within established control limits for accuracy (%Recovery) and precision (RPD). Deviations from this may be the results of matrix effects, which are confirmed by passing LCSs.

13.1.3.2. No specific corrective actions are required in the evaluation of the MS/MSD results provided that the batch LCS is in control. Analysts should use sound judgement in accepting MS/MSD results that are not within control limits, especially if the LCS results are borderline.

13.2. Sample result evaluation

13.2.1. Carryover – When a sample has a high response for a compound, there is a real possibility that some of the sample may carry over into the sample analyzed immediately afterward.

13.2.1.1. If a sample analyzed after a sample with high concentrations has negative results, carryover did not occur.

13.2.1.2. If a sample analyzed after a sample with high concentrations has positive results for the same analytes, or if the chromatographic profile resembles the previous sample, the results are questionable. This sample must be reanalyzed under conditions in which carryover can be confirmed to not have occurred.

13.2.2. Dilutions

13.2.2.1. If the response for any compound exceeds the working range of the analytical system, a dilution of the extract is prepared and analyzed. An appropriate dilution should be in the upper half of the calibration range.

13.2.2.2. If the initial diluted run has no hits or hits below 20% of the calibration range and the matrix allows for analysis at a lesser dilution, then the sample must be reanalyzed at a dilution targeted to bring the largest hit in the upper half of the calibration range.

14. CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

14.1. Method Blank.

14.1.1. The samples in the batch associated to the defective method blank are evaluated.

14.1.1.1. If the analyte found in the method blank is confirmed to not be present in any of the associated samples at any level, the contamination did not affect those samples. This represents a non-impact situation and no specific corrective action is taken. A nonconformance memo is written to notify project management of the situation for evaluation against project requirements.

14.1.1.2. If the analyte found in the method blank is confirmed to be present in one or more of the associated samples, the concentrations are compared.

14.1.1.2.1. If the concentration in the method blank exceeded 10% of concentration found in one or more samples, the prescribed corrective action is to re-analyze all affected samples.

14.1.1.2.2. If the concentration in the method blank was less than 10% of the concentration found in one or more samples, the sample can be reported by qualifying the affected analytes. A nonconformance memo is written to notify project management of the situation for evaluation against project requirements.

14.1.2. If the analyte found in the method blank is not one of the common laboratory contaminants (acetone, methylene chloride, 2-butanone), the analytical system must be placed out of service until the source of the problem is identified and corrected.

14.2. Laboratory control sample

14.2.1. If any control analyte is out of control for accuracy (% Recovery), the associated samples are evaluated.

14.2.1.1. If the recovery is biased high and the associated samples have no positive results for that analyte, a non-impact situation ensues. A nonconformance memo is written to notify project management of the situation for evaluation against project requirements.

14.2.1.2. If the recovery is biased low and the associated samples have positive results for that analyte, a minimal impact situation ensues. A nonconformance memo is written to notify project management of the situation for evaluation against project requirements.

14.2.1.3. If the recovery is biased high and the associated samples have positive results for that analyte, the prescribed corrective action is reanalysis of the affected sample(s).

14.2.1.4. If the recovery is biased low and the samples have no positive results for that analyte, the prescribed corrective action is reanalysis of the affected sample(s).

14.2.2. If any control analyte is out of control for precision (RPD), the associated samples are evaluated. All decisions about corrective action(s) are made in consultation with the project manager.

14.2.2.1. If there are no positive results in one or more samples, a non-impact situation ensues for those samples. A nonconformance memo is written to notify project management of the situation for evaluation against project requirements.

14.2.2.2. If there are positive results for one or more analytes, the likelihood of poor reproducibility increases and corrective action must be evaluated. A nonconformance memo is written to notify project

management of the situation for a project decision on whether the affected sample(s) should be reanalyzed.

15. CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

15.1. Method blanks

15.1.1. If there is insufficient sample to perform re-analysis, the analyst must notify the project manager for consultation with the client. In this situation, all associated samples are flagged with a "B" qualifier and appropriate comments in the narrative.

15.2. LCS

15.2.1. If the batch is not re-extracted and reanalyzed, the reasons for accepting the batch must be clearly presented in the project records and the report. Acceptable matrix spike recoveries are not sufficient justification to preclude re-extraction of the batch.

15.2.2. If re-extraction and reanalysis of the batch is not possible due to limited sample volume or other constraints, the LCS is reported, all associated samples are flagged, and appropriate comments are made in a narrative to provide further documentation.

15.3. Insufficient sample

15.3.1. If there is insufficient sample to repeat the analysis, the project manager is notified via NCM for consultation with the client.

16. METHOD PERFORMANCE

16.1. Method Detection Limit

Each laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in QA Policy No. QA-005, Determination of Method Detection Limits for Chemical Tests. When non-standard compounds are analyzed at client request, lesser requirements are possible with client agreement. At a minimum, a standard at the reporting limit must be analyzed to demonstrate the capability of the method.

16.2. Initial Demonstration

16.2.1. Each laboratory must make a one time initial demonstration of capability for each individual method. Demonstration of capability for both soils and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest. The QC check sample is made up at 20 µg/L. (Some compounds will be at higher levels, refer to the calibration standard levels for guidance.)

16.2.2. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation.

16.2.3. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. The %RSD should be $\leq$ 15% for each analyte, and the % recovery should be within 80-120%.

16.2.4. If any analyte does not meet the acceptance criteria, check the acceptance limits in the reference methods. If the recovery or precision is outside the limits in the reference methods, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

16.2.5. Training Qualification

The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

17. POLLUTION PREVENTION

This procedure will be carried out in a manner consistent with all applicable federal state and local regulations regarding pollution control and prevention. Specific controls due to the accidental release of hazardous materials can be found in the Chemical Hygiene Plan and facility attachments.

18. WASTE MANAGEMENT

Waste generated in this procedure will be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Coordinator should be contacted if additional information is required.

19. REFERENCES

19.1. SW846, *Test Methods for Evaluating Solid Waste,* Third Edition, Gas Chromatography/Mass Spectrometry for Volatile Organics, Method 8260B, Update III, December 1996

19.2. Associated SOPs

19.2.1. STL-QA-0031, "VOA Holding Blank Analysis"

20. CLARIFICATIONS, MODIFICATIONS AND ADDITIONS TO REFERENCE METHOD

20.1. Modifications from the reference method

20.1.1. Ion 119 is used as the quantitation ion for chlorobenzene-d5 for 25 mL purge tests.

20.1.2. A retention time window of 0.2 minutes is used for all components, since some data systems do not have the capability of using the relative retention time units specified in the reference method.

20.1.3. The quantitation and qualifier ions for some compounds have been changed from those recommended in SW-846 in order to improve the reliability of qualitative identification.

20.1.4. SW-846 recommends that a curve be used for any analytes with %RSD of the response factors > 15%. However, some industry standard data systems and forms generation software cannot report this data with the necessary information for data validation. In addition most software available does not allow weighting of the curve. Unweighted curves may exhibit serious errors in quantitation at the low end, resulting in possible false positives or false negatives. Therefore, this SOP allows the use of average response factors if the average %RSD for all compounds is $\leq$ 15%.

20.2. Modifications from previous revision

This SOP has been substantially revised to reflect the changes included in Update III to SW-846. Directions for method 524.2 and method 624 have also been added.

20.3. Facility specific SOPs

Each facility shall attach a list of facility-specific SOPs or approved attachments (if applicable) which are required to implement this SOP or which are used in conjunction with this SOP. If no facility specific SOPs or amendments are to be attached, a statement must be attached specifying that there are none.

20.4. Flow diagrams

20.4.1. Initial Demonstration and MDL

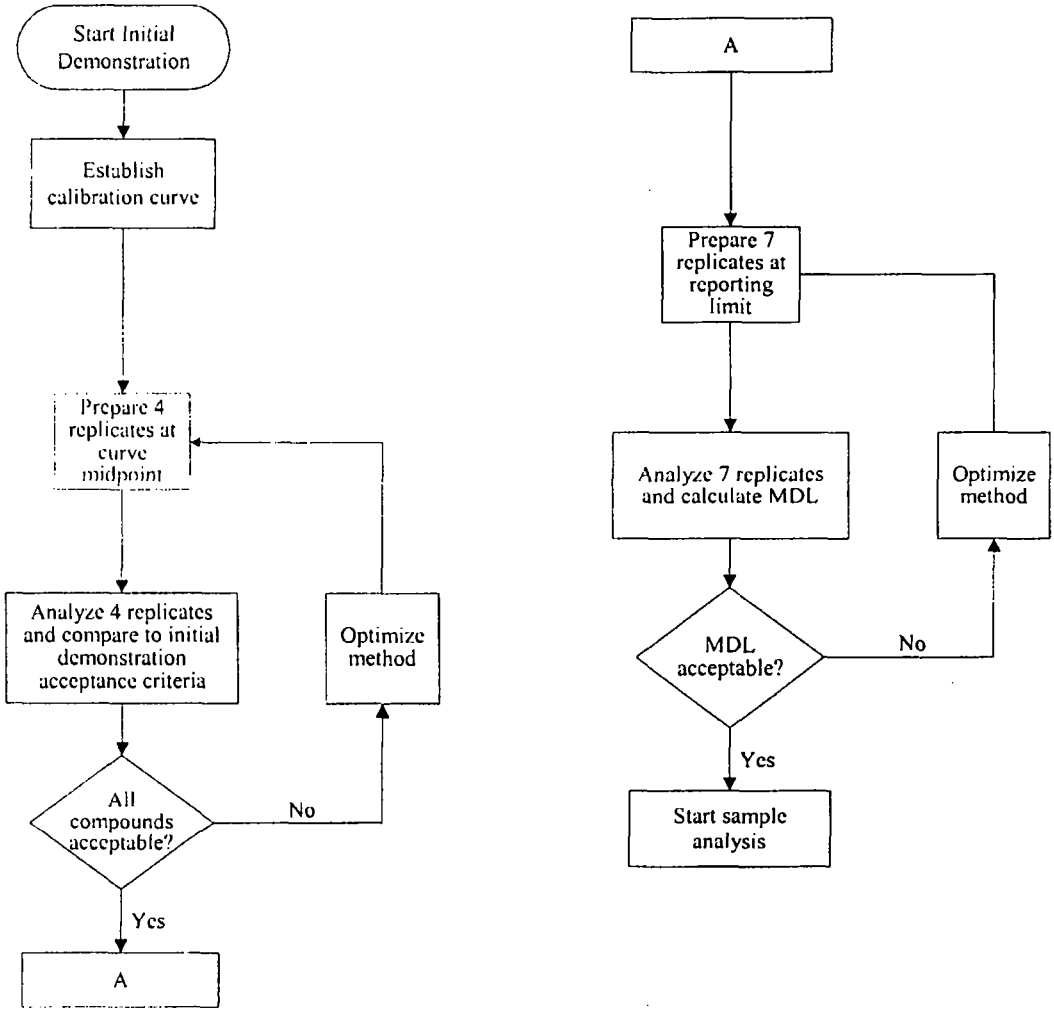


Table 1

Primary Standard and Reporting Limits

Compound	CAS Number	Reporting Limits[1]			
		5 mL Water µg/L	25 mL water µg/L	Low soil µg/kg	Med. Soil µg/kg
Dichlorodifluoromethane	75-71-8	10	2	10	500
Chloromethane	74-87-3	10	2	10	500
Bromomethane	74-83-9	10	2	10	500
Vinyl chloride	75-01-4	10	2	10	500
Chloroethane	75-00-3	10	2	10	500
Trichlorofluoromethane	75-69-4	10	2	10	500
Acrolein	107-02-8	100	20	100	5000
Acetone	67-64-1	20	10	20	1000
Trichlorotrifluoroethane	76-13-1	5	1	5	250
Ethanol	64-17-5	500	200	500	25,000
Iodomethane	74-88-4	5	1	5	250
Carbon disulfide	75-15-0	5	1	5	250
Methylene chloride	75-09-2	5	1	5	250
tert-Butyl alcohol	75-65-0	200	50	200	10,000
1,1-Dichloroethene	75-35-4	5	1	5	250
1,1-Dichloroethane	75-34-3	5	1	5	250
trans-1,2-Dichloroethene	156-60-5	2.5	0.5	2.5	125
Acrylonitrile	107-13-1	100	20	100	5000
Methyl *tert*-butyl ether (MTBE)	1634-04-4	20	5	20	1000
Hexane	110-54-3	5	1	5	250
cis-1,2-Dichloroethene	156-59-2	2.5	0.5	2.5	125
1,2-Dichloroethene (Total)	540-59-0	5	1	5	250
Tetrahydrofuran	109-99-9	20	5	20	1000
Chloroform	67-66-3	5	1	5	250
1,2-Dichloroethane	107-06-2	5	1	5	250
Dibromomethane	74-95-3	5	1	5	250
2-Butanone	78-93-3	20	5	20	1000
1,4-Dioxane	123-91-1	500	200	500	25000
1,1,1-Trichloroethane	71-55-6	5	1	5	250
Carbon tetrachloride	56-23-5	5	1	5	250
Bromodichloromethane	75-27-4	5	1	5	250
1,2-Dichloropropane	78-87-5	5	1	5	250
cis-1,3-Dichloropropene	10061-01-5	5	1	5	250
Trichloroethene	79-01-6	5	1	5	250
Dibromochloromethane	124-48-1	5	1	5	250

Table 1

Primary Standard and Reporting Limits

Compound	CAS Number	Reporting Limits[1] 5 mL Water µg/L	25 mL water µg/L	Low soil µg/kg	Med. Soil µg/kg
1,2-Dibromoethane	106-93-4	5	1	5	250
1,2,3-Trichloropropane	96-18-4	5	1	5	250
1,1,2-Trichloroethane	79-00-5	5	1	5	250
Benzene	71-43-2	5	1	5	250
Ethylmethacrylate	97-63-2	5	1	5	250
trans-1,3-Dichloropropene	10061-02-6	5	1	5	250
Bromoform	75-25-2	5	1	5	250
4-Methyl-2-pentanone	108-10-1	20	5	20	1000
2-Hexanone	591-78-6	20	5	20	1000
Tetrachloroethene	127-18-4	5	1	5	250
Toluene	108-88-3	5	1	5	250
1,1,2,2-Tetrachloroethane	79-34-5	5	1	5	250
2-Chloroethyl vinyl ether	110-75-8	N/A[2]	N/A	50	1000
Vinyl acetate	108-05-4	10	2	10	500
Chlorobenzene	108-90-7	5	1	5	250
Ethylbenzene	100-41-4	5	1	5	250
Styrene	100-42-5	5	1	5	250
t-1,4-Dichloro-2-butene	110-57-6	5	1	5	250
m and p Xylenes		2.5	0.5	2.5	125
o-xylene	95-47-6	2.5	0.5	2.5	125
Total xylenes	1330-20-7	5	1	5	250
1,3-Dichlorobenzene	541-73-1	5	1	5	250
1,4-Dichlorobenzene	106-46-7	5	1	5	250
1,2-Dichlorobenzene	95-50-1	5	1	5	250

[1] Reporting limits listed for soil/sediment are based on wet weight. The reporting limits calculated by the laboratory for soil/sediment, calculated on dry weight basis, will be higher.

[2] 2-Chloroethyl vinyl ether cannot be reliably recovered from acid preserved samples

Table 2

Primary Standard Calibration Levels, 5 mL purge[1]

Compound	Calibration Level ug/L					
	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6
1,2-Dichloroethane-d4 (Surrogate)	5	10	20	50	100	200
Toluene-d8 (Surrogate)	5	10	20	50	100	200
4-Bromofluorobenzene (Surrogate)	5	10	20	50	100	200
Dichlorodifluoromethane	5	10	20	50	100	200
Chloromethane	5	10	20	50	100	200
Bromomethane	5	10	20	50	100	200
Vinyl chloride	5	10	20	50	100	200
Chloroethane	5	10	20	50	100	200
Trichlorofluoromethane	5	10	20	50	100	200
Acrolein	50	100	200	500	1000	2000
Acetone	5	10	20	50	100	200
Trichlorotrifluoroethane	5	10	20	50	100	200
Ethanol	500	1000	2000	5000	10000	20000
Iodomethane	5	10	20	50	100	200
Carbon disulfide	5	10	20	50	100	200
Methylene chloride	5	10	20	50	100	200
tert-Butyl alcohol	100	200	400	1,000	2,000	4,000
1,1-Dichloroethene	5	10	20	50	100	200
1,1-Dichloroethane	5	10	20	50	100	200
trans-1,2-Dichloroethene	5	10	20	50	100	200
Acrylonitrile	50	100	200	500	1,000	2,000
Methyl *tert*-butyl ether (MTBE)	5	10	20	50	100	200
Hexane	5	10	20	50	100	200
cis-1,2-Dichloroethene	5	10	20	50	100	200
Tetrahydrofuran	5	10	20	50	100	200
Chloroform	5	10	20	50	100	200
1,2-Dichloroethane	5	10	20	50	100	200
Dibromomethane	5	10	20	50	100	200
2-Butanone	5	10	20	50	100	200
1,4-Dioxane	250	500	1000	2,500	5,000	10,000
1,1,1-Trichloroethane	5	10	20	50	100	200
Carbon tetrachloride	5	10	20	50	100	200
Bromodichloromethane	5	10	20	50	100	200
1,2-Dichloropropane	5	10	20	50	100	200
cis-1,3-Dichloropropene	5	10	20	50	100	200

Table 2

Primary Standard Calibration Levels, 5 mL purge[1]

Compound	Calibration Level ug/L					
	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6
Trichloroethene	5	10	20	50	100	200
Dibromochloromethane	5	10	20	50	100	200
1,2-Dibromoethane	5	10	20	50	100	200
1,2,3-Trichloropropane	5	10	20	50	100	200
1,1,2-Trichloroethane	5	10	20	50	100	200
Benzene	5	10	20	50	100	200
Ethylmethacrylate	5	10	20	50	100	200
trans-1,3-Dichloropropene	5	10	20	50	100	200
Bromoform	5	10	20	50	100	200
4-Methyl-2-pentanone	5	10	20	50	100	200
2-Hexanone	5	10	20	50	100	200
Tetrachloroethene	5	10	20	50	100	200
Toluene	5	10	20	50	100	200
1,1,2,2-Tetrachloroethane	5	10	20	50	100	200
2-Chloroethyl vinyl ether	10	20	40	100	200	400
Vinyl acetate	5	10	20	50	100	200
Chlorobenzene	5	10	20	50	100	200
Ethylbenzene	5	10	20	50	100	200
Styrene	5	10	20	50	100	200
t-1,4-Dichloro-2-butene	5	10	20	50	100	200
m and p Xylenes	10	20	40	100	200	400
o-xylene	5	10	20	50	100	200
1,3-Dichlorobenzene	5	10	20	50	100	200
1,4-Dichlorobenzene	5	10	20	50	100	200
1,2-Dichlorobenzene	5	10	20	50	100	200

[1] **Levels for 25 mL purge are 5 times lower in all cases**

Table 3

Appendix IX Standard and Reporting Limits, 5 mL purge[1]

Compound	CAS Number	Reporting Limits			
		5 mL Water µg/L	25 mL water µg/L	Low Soil µg/kg	Medium Soil µg/mL
Allyl Chloride	107-05-1	10	2	10	500
Acetonitrile	75-05-8	100	20	100	5000
Dichlorofluoromethane		10	2	10	500
Isopropyl ether	108-20-3	10	2	10	500
Chloroprene	126-99-8	5	1	5	250
n-Butanol	71-36-3	200	50	200	10,000
Propionitrile	107-12-0	20	4	20	1000
Methacrylonitrile	126-98-7	10	2	5	250
Isobutanol	78-83-1	200	50	200	10,000
Methyl methacrylate	80-62-6	5	1	5	250
1,1,1,2-Tetrachloroethane	630-20-6	5	1	5	250
1,2-Dibromo-3-chloropropane	96-12-8	10	2	10	500
Ethyl ether	60-29-7	10	2	10	500
Ethyl Acetate	141-78-6	20	4	20	1,000
2-Nitropropane	79-46-9	10	2	10	500
Cyclohexanone	108-94-1	N/A[2]	N/A[2]	N/A[2]	N/A[2]
Isopropylbenzene	98-82-8	5	1	5	250

[1] **Levels for 25 mL purge are 5 times lower in all cases**

[2] Cyclohexanone decomposes to 1,1-dimethoxycyclohexane in methanolic solution. Reporting limits cannot be accurately determined.

Table 4

Appendix IX Standard Calibration Levels, µg/L

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6
Allyl Chloride	5	10	20	50	100	200
Acetonitrile	50	100	200	500	1,000	2,000
Dichlorofluoromethane	5	10	20	50	100	200
Isopropyl ether	5	10	20	50	100	200
Chloroprene	5	10	20	50	100	200
n-Butanol	100	200	400	1,000	2,000	4,000
Propionitrile	10	20	40	100	200	400
Methacrylonitrile	5	10	20	50	100	200
Isobutanol	100	200	400	1,000	2,000	4,000
Methyl methacrylate	5	10	20	50	100	200
1,1,1,2-Tetrachloroethane	5	10	20	50	100	200
1,2-Dibromo-3-chloropropane	10	20	40	100	200	400
Ethyl ether	5	10	20	50	100	200
Ethyl Acetate	10	20	40	100	200	400
2-Nitropropane	10	20	40	100	200	400
Cyclohexanone	50	100	200	500	1,000	2,000
Isopropylbenzene	5	10	20	50	100	200

Table 5

Reportable Analytes for Standard Tests, Primary Standard

Compound	CAS Number	Standard List	TCLP	TCL	Appendix IX	UTS
Dichlorodifluoromethane	75-71-8				X	X
Chloromethane	74-87-3	X		X	X	X
Bromomethane	74-83-9	X		X	X	X
Vinyl chloride	75-01-4	X	X	X	X	X
Chloroethane	75-00-3	X		X	X	X
Trichlorofluoromethane	75-69-4				X	X
Acrolein	107-02-8				X	X
Acetone	67-64-1	X		X	X	X
Trichlorotrifluoroethane	76-13-1					X
Ethanol	64-17-5					
Iodomethane	74-88-4				X	X
Carbon disulfide	75-15-0	X		X	X	X
Methylene chloride	75-09-2	X		X	X	X
tert-Butly alcohol	75-65-0					
1,1-Dichloroethene	75-35-4	X	X	X	X	X
1,1-Dichloroethane	75-34-3	X		X	X	X
trans-1,2-Dichloroethene	156-60-5	X		X	X	X
Total dichloroethene		X		X	X	X
Acrylonitrile	107-13-1				X	X
Methyl *tert*-butyl ether (MTBE)	1634-04-4					
Hexane	110-54-3					
cis-1,2-Dichloroethene	156-59-2	X		X		
Tetrahydrofuran	109-99-9					
Chloroform	67-66-3	X	X	X	X	X
1,2-Dichloroethane	107-06-2	X	X	X	X	X
Dibromomethane	74-95-3				X	X
2-Butanone	78-93-3	X	X	X	X	X
1,4-Dioxane	123-91-1				X	X
1,1,1-Trichloroethane	71-55-6	X		X	X	X
Carbon tetrachloride	56-23-5	X	X	X	X	X
Bromodichloromethane	75-27-4	X		X	X	X
1,2-Dichloropropane	78-87-5	X		X	X	X
cis-1,3-Dichloropropene	10061-01-5	X		X	X	X

Table 5

Reportable Analytes for Standard Tests, Primary Standard

Compound	CAS Number	Standard List	TCLP	TCL	Appendix IX	UTS
Trichloroethene	79-01-6	X	X	X	X	X
Dibromochloromethane	124-48-1	X		X	X	X
1,2-Dibromoethane	106-93-4				X	X
1,2,3-Trichloropropane	96-18-4				X	X
1,1,2-Trichloroethane	79-00-5	X		X	X	X
Benzene	71-43-2	X	X	X	X	X
Ethylmethacrylate	97-63-2				X	X
trans-1,3-Dichloropropene	10061-02-6	X		X	X	X
Bromoform	75-25-2	X		X	X	X
4-Methyl-2-pentanone	108-10-1	X		X	X	X
2-Hexanone	591-78-6	X		X	X	
Tetrachloroethene	127-18-4	X	X	X	X	X
Toluene	108-88-3	X		X	X	X
1,1,2,2-Tetrachloroethane	79-34-5	X		X	X	X
2-Chloroethyl vinyl ether	110-75-8					
Vinyl acetate	108-05-4				X	
Chlorobenzene	108-90-7	X	X	X	X	X
Ethylbenzene	100-41-4	X		X	X	X
Styrene	100-42-5	X		X	X	
t-1,4-Dichloro-2-butene	110-57-6				X	
m and p Xylenes		X		X	X	X
o-xylene	95-47-6	X		X	X	X
Total xylenes	1330-20-7	X		X	X	X
1,3-Dichlorobenzene	541-73-1					
1,4-Dichlorobenzene	106-46-7					
1,2-Dichlorobenzene	95-50-1					

Table 6

Reportable Analytes for Standard Tests, Appendix IX standard

Compound	Number	Standard List	TCLP	TCL	Appendix IX	UTS
Allyl Chloride	107-05-1				X	
Acetonitrile	75-05-8				X	X
Dichlorofluoromethane	75-43-4					
Isopropyl ether	108-20-3					
Chloroprene	126-99-8				X	
n-Butanol	71-36-3					
Propionitrile	107-12-0				X	
Methacrylonitrile	126-98-7				X	X
Isobutanol	78-83-1				X	X
Methyl methacrylate	80-62-6				X	X
1,1,1,2-Tetrachloroethane	630-20-6				X	X
1,2-Dibromo-3-chloropropane	96-12-8				X	X
Ethyl ether	60-29-7					X
Ethyl Acetate	141-78-6					X
2-Nitropropane	79-46-9					
Cyclohexanone	108-94-1					X
Isopropylbenzene	98-82-8					

Table 7

Internal Standards

	Standard Concentration µg/mL	Quantitation ion (5 mL purge)	Quantitation ion (25 mL purge)
Fluorobenzene	25	96	96
Chlorobenzene-d5	25	117	119
1,4-Dichlorobenzene-d4	25	152	152

Notes:

1) 10 µL of the internal standard is added to the sample. This results in a concentration of each internal in the sample of 50µg/L for a 5 mL purge or 10 µg/L for a 25 mL purge.
2) Except for medium level soils, the surrogate and internal standards may be combined in one solution.

Table 8

Surrogate Standards

Surrogate Compounds	Standard Concentration µg/mL
1,2-Dichloroethane-d_4	25
Dibromofluoromethane	25
Toluene-d_8	25
4-Bromofluorobenzene	25

Notes:

1) 10 µL of the surrogate standard is added to the sample. This results in a concentration of each surrogate in the sample of 50µg/L for a 5 mL purge or 10 µg/L for a 25 mL purge.
2) Except for medium level soils, the surrogate and internal standards may be combined in one solution.
3) Recovery limits for surrogates are generated from historical data and are maintained by the QA department.

Table 9

Matrix Spike / LCS Compounds

Compound	Standard Concentration µg /mL
1,1-Dichloroethene	25
Trichloroethene	25
Toluene	25
Benzene	25
Chlorobenzene	25

Notes:

1) 10 µL of the standard is added to the LCS or matrix spiked sample. This results in a concentration of each spike analyte in the sample of 50µg/L for a 5 mL purge or 10 µg/L for a 25 mL purge.
2) Recovery and precision limits for LCS and MS/MSD are generated from historical data and are maintained by the QA department.
3) Full analyte spikes may also be used at the laboratories' option or at client request.

Table 10

BFB Key Ion Abundance Criteria

Mass	Ion Abundance Criteria
50	15% to 40% of Mass 95
75	30% to 60% of Mass 95
95	Base Peak, 100% Relative Abundance
96	5% to 9% of Mass 95
173	Less Than 2% of Mass 174
174	Greater Than 50% of Mass 95
175	5% to 9% of Mass 174
176	Greater Than 95%, But Less Than 101% of Mass 174
177	5% to 9% of Mass 176

Table 11

SPCC Compounds and Minimum Response Factors

Compound	8240B Min. RF	8260B Min. RF
Chloromethane	0.300	0.100
1,1-Dichloroethane	0.300	0.100
Bromoform	>0.100	>0.100
1,1,2,2-Tetrachloroethane	0.300	0.300
Chlorobenzene	0.300	0.300

Table 12

CCC compounds

Compound	Max. %RSD from Initial Calibration	Max. %D for continuing calibration
Vinyl Chloride	≤30.0	≤20.0
1,1-Dichloroethene	≤30.0	≤20.0
Chloroform	≤30.0	≤20.0
1,2-Dichloropropane	≤30.0	≤20.0
Toluene	≤30.0	≤20.0
Ethylbenzene	≤30.0	≤20.0

Table 13

Characteristic ions

Compound	Primary*	Secondary	Tertiary
1,2-Dichloroethane-d_4 (Surrogate)	65	102	
Dichlorodifluoromethane	85	87	50, 101,103
Chloromethane	50	52	49
Vinyl chloride	62	64	61
Bromomethane	94	96	79
Chloroethane	64	66	49
Trichlorofluoromethane	101	103	66
1,1-Dichloroethene	96	61	98
Acrolein	56	55	58
Iodomethane	142	127	141
Carbon disulfide	76	78	
Trichlorotrifluoroethane	151	101	153

Table 13

Characteristic ions

Compound	Primary*	Secondary	Tertiary
Ethanol	45	46	
Acetone	43	58	
Methylene chloride	84	49	51, 86
tert-Butyl alcohol	59	74	
trans-1,2-Dichloroethene	96	61	98
Acrylonitrile	53	52	51
Methyl *tert* butyl ether	73		
Hexane	57	43	
1,1-Dichloroethane	63	65	83
cis-1,2-Dichloroethene	96	61	98
2-Butanone	43	72**	
Tetrahydrofuran	42	71	
Chloroform	83	85	47
1,2-Dichloroethane	62	64	98
Dibromomethane	93	174	95, 172, 176
1,4-Dioxane	88	58	
Vinyl acetate	43	86	
1,1,1-Trichloroethane	97	99	117
Carbon tetrachloride	117	119	121
Benzene	78	52	77
Trichloroethene	130	95	97, 132
1,2-Dichloropropane	63	65	41
Bromodichloromethane	83	85	129
2-Chloroethyl vinyl ether	63	65	106
cis-1,3-Dichloropropene	75	77	39
trans-1,3-Dichloropropene	75	77	39
1,1,2-Trichloroethane	97	83	85, 99
Chlorodibromomethane	129	127	131
Bromoform	173	171	175, 252
1,2,3-Trichloropropane	75	110	77, 112, 97
Toluene-d_8 (Surrogate)	98	70	100
4-Bromofluorobenzene (Surrogate)	95	174	176
Toluene	91	92	65
4-Methyl-2-pentanone	43	58	57, 100
Tetrachloroethene	164	166	131
Ethyl methacrylate	69	41	99, 86, 114
2-Hexanone	43	58	57, 100

Table 13

Characteristic ions

Compound	Primary*	Secondary	Tertiary
Chlorobenzene	112	114	77
Ethylbenzene	106	91	
Xylenes	106	91	
Styrene	104	103	78, 51, 77
Dichlorobenzene (all isomers)	146	148	111
trans 1,4-Dichloro-2-butene	53	75	89, 77, 124
1,1,2,2-Tetrachloroethane	83	85	131, 133
Allyl Chloride	76	41	78
Acetonitrile	40	41	
Dichlorofluoromethane	67	69	
Isopropyl ether	87	59	45
Chloroprene	53	88	90
n-Butanol	56	41	42
Propionitrile	54	52	55
Methacrylonitrile	41	67	52
Isobutanol	41	43	74
Methyl methacrylate	41	69	100
1,1,1,2-Tetrachloroethane	131	133	119
1,2-Dibromo-3-chloropropane	157	155	75
Ethyl ether	59	74	
Ethyl Acetate	43	88	61
2-Nitropropane	41	43	46
Cyclohexanone	55	42	98
Isopropylbenzene	105	120	

* The primary ion should be used for quantitation unless interferences are present, in which case a secondary ion may be used.
** m/z 43 may be used for quantitation of 2-Butanone, but m/z 72 must be present for positive identification.

21. APPENDIX A

This Appendix A lists modifications to the main body of the SOP that are necessary for analysis of drinking water by method 524.2.

21.1. A target analyte list based on the list in method 524.2 is frequently requested for analysis by method 8260B. STL's standard analyte list for this test, and the internal and surrogate standards used, are listed in Tables A-1 to A-4 below. In all other respects the method is as described in the main body of this SOP. Note that this without the modifications listed in Section 22, the method is ***not*** appropriate for drinking water analysis by method 524.2.

22. MODIFICATIONS REQUIRED FOR DRINKING WATER ANALYSIS BY METHOD 524.2

22.1. This method can be applied to surface water, ground water and drinking water.

22.2. Purge sample volume is normally 25 mL, but lesser volumes may be used if adequate sensitivity is obtained.

22.3. Sample concentrations are calculated using initial calibration curve.

22.4. Only one internal standard -- Fluorobenzene -- is used for this method, and therefore all target analytes are assigned to it.

22.5. A maximum of 25 ng of BFB is used for tuning for method 524.2

22.6. BFB tuning criteria for mass 75 are 30-80% of mass 95.

22.7. The recovery limits for the initial demonstration of capability are 80-120% with %RSD less than 20%.

22.8. Initial calibration curve requirements:

22.8.1. The number of calibration standards depends on the calibration range used. For a range of up to a factor of 20 (e.g. 1µg/L - 20µg/L) a minimum of three standards are necessary. For a factor of up to 50 four standards are necessary, and for a factor of up to 100 five standards are necessary.

22.8.2. All target compounds must have RSD ≤ 20%.

22.8.3. If this requirement can not be met, a regression curve must be constructed for the non-compliant compounds. There is no correlation coefficient requirement for the regression curve.

22.9. Continuing calibration verification (CCV) requirements:

22.9.1. All target compounds must have %D ≤ 30%.

22.9.2. The internal standards in each CCV must be over 70% of the abundance found in the CCV analysis immediately preceding it *and* over 50% of the calibration point in the initial calibration curve whose concentration matches that of the CCV.

22.9.3. The same analysis run may be used to satisfy the requirements for an LCS (also known as a laboratory fortified blank, LFB) and a continuing calibration verification. The LCS/CCV does not need to be a second source standard.

22.10. Method clarifications, modifications and additions

22.10.1. Section 7.1 of the source method requires that the trap packing materials be Tenax GC, Methyl silicone, silica gel and coconut charcoal. STL routinely employs the Supelco VOCARB 3000, which consists of Carbopack B and Carboxen 1000 and 1001.

22.10.2. Section 7.8.2 of the source method requires that each calibration standard be prepared by diluting the appropriate volume of the working standard with organic-free water adjusted to pH < 2 in a volumetric flask. STL prepares calibration standards by diluting the appropriate volume of the working standard with organic-free water in the gas-tight syringe that will be used to inject the sample into the purge and trap device.

22.10.3. Sections 9.8 and 9.9 of the source method require that duplicate spiked blanks and a second-source initial calibration verification standard be analyzed at least quarterly. Since some STL do not normally analyze drinking waters samples, these QC samples will be analyzed only during the conduct of projects that require this method.

Table A-1

8260 Drinking Water List Standard and Reporting Limits

Compound	CAS Number	Reporting Limits[1]			
		5 mL water µg/L	25 mL water µg/L	Low soil µg/kg	Med. Soil µg/kg
Dichlorodifluoromethane	75-71-8	10	2	10	500
Chloromethane	74-87-3	10	2	10	500
Bromomethane	74-83-9	10	2	10	500
Vinyl chloride	75-01-4	10	2	10	500
Chloroethane	75-00-3	10	2	10	500
Trichlorofluoromethane	75-69-4	10	2	10	500
Acetone[1]	67-64-1	20	10	20	1000
Methylene chloride	75-09-2	5	2	5	250
1,1-Dichloroethene	75-35-4	5	1	5	250
1,1-Dichloroethane	75-34-3	5	1	5	250
Trans-1,2-Dichloroethene	156-60-5	2.5	0.5	2.5	125
Methyl *tert*-butyl ether (MTBE)[1]	1634-04-4	20	5	20	250
2,2-Dichloropropane	590-20-7	5	1	5	250
Cis-1,2-Dichloroethene	156-59-2	2.5	0.5	2.5	125
1,2-Dichloroethene (Total)	540-59-0	5	1	5	250
Chloroform	67-66-3	5	1	5	250
Bromochloromethane	74-97-5	5	1	5	250
1,2-Dichloroethane	107-06-2	5	1	5	250
Dibromomethane	74-95-3	5	1	5	250
2-Butanone[1]	78-93-3	20	5	20	1000
1,1,1-Trichloroethane	71-55-6	5	1	5	250
Carbon tetrachloride	56-23-5	5	1	5	250
1,1-Dichloropropene	563-58-6	5	1	5	250
Bromodichloromethane	75-27-4	5	1	5	250
1,2-Dichloropropane	78-87-5	5	1	5	250
1,3-Dichloropropane	142-28-9	5	1	5	250
Cis-1,3-Dichloropropene	10061-01-5	5	1	5	250
Trichloroethene	79-01-6	5	1	5	250
Dibromochloromethane	124-48-1	5	1	5	250
1,2-Dibromoethane	106-93-4	5	1	5	250
1,2,3-Trichloropropane	96-18-4	5	1	5	250
1,1,2-Trichloroethane	79-00-5	5	1	5	250
Benzene	71-43-2	5	1	5	250
Trans-1,3-Dichloropropene	10061-02-6	5	1	5	250

Table A-1

8260 Drinking Water List Standard and Reporting Limits

Compound	CAS Number	Reporting Limits[1] 5 mL water µg/L	25 mL water µg/L	Low soil µg/kg	Med. Soil µg/kg
Bromoform	75-25-2	5	1	5	250
4-Methyl-2-pentanone[1]	108-10-1	20	5	20	1000
2-Hexanone[1]	591-78-6	20	5	20	1000
Tetrachloroethene	127-18-4	5	1	5	250
Toluene	108-88-3	5	1	5	250
1,1,2,2-Tetrachloroethane	79-34-5	5	1	5	250
Chlorobenzene	108-90-7	5	1	5	250
1,1,1,2-Terrachloroethane	630-20-6	5	1	5	250
Ethylbenzene	100-41-4	5	1	5	250
Styrene	100-42-5	5	1	5	250
m and p Xylenes		2.5	0.5	2.5	125
o-xylene	95-47-6	2.5	0.5	2.5	125
Total xylenes	1330-20-7	5	1	5	250
Isopropylbenzene	98-82-8	5	1	5	250
Bromobenzene	108-86-1	5	1	5	250
n-Propylbenzene	103-65-1	5	1	5	250
2-Chlorotoluene	95-49-8	5	1	5	250
4-Chlorotoluene	106-43-4	5	1	5	250
1,3,5-Trimethylbenzene	108-67-8	5	1	5	250
tert-Butylbenzene	98-06-6	5	1	5	250
1,2,4-Trimethylbenzene	95-63-6	5	1	5	250
sec-butylbenzene	135-98-8	5	1	5	250
1,3-Dichlorobenzene	541-73-1	5	1	5	250
1,4-Dichlorobenzene	106-46-7	5	1	5	250
1,2-Dichlorobenzene	95-50-1	5	1	5	250
4-Isopropyltoluene	99-87-6	5	1	5	250
n-Butylbenzene	104-51-8	5	1	5	250
1,2-Dibromo-3-chloropropane	96-12-8	5	1	5	250
1,2,4-Trichlorobenzene	120-82-1	5	1	5	250
Naphthalene	91-20-3	5	1	5	250
Hexachlorobutadiene	87-68-3	5	1	5	250
1,2,3-Trichlorobenzene	87-61-6	5	1	5	250

[1] Not included on the method 524.2 analyte list, but includes in the calibration standard as an add on frequently requested by method 8260B.

Table A-2

Internal Standards, Method 8260A Drinking water list

	Standard Concentration µg/mL	Quantitation ion
Fluorobenzene	25	96
Chlorobenzene-d5	25	119
1,4-Dichlorobenzene-d4	25	152

Notes:
1) Fluorobenzene only is used for method 524.2
2) 10 µL of the internal standard is added to the sample. This results in a concentration of each internal in the sample of 50µg/L for a 5 mL purge or 10 µg/L for a 25 mL purge.
3) Except for medium level soils, the surrogate and internal standards may be combined in one solution.

Table A-3

Surrogate Standards, Drinking water list

Surrogate Compounds	Standard Concentration µg/mL
1,2-Dichloroethane-d_4[1]	25
Dibromofluoromethane[1]	25
Toluene-d_8[1]	25
1,2-Dichlorobenzene-d4[2]	25
4-Bromofluorobenzene[1,2]	25

[1] 8260B surrogate
[2] 524.2 surrogate

Notes:
1) 10 µL of the surrogate standard is added to the sample. This results in a concentration of each surrogate in the sample of 50µg/L for a 5 mL purge or 10 µg/L for a 25 mL purge.
2) Except for medium level soils, the surrogate and internal standards may be combined in one solution.

Table A-4

Drinking water list Standard: Calibration Levels

Compound	Level 1		Level 2		Level 3		Level 4		Level 5	
	5 mL	25 mL	5 mL	25 mL	5 mL	25 mL	5 mL	25 mL	5 mL	25 mL
Dichlorodifluoromethane	10	2	40	10	100	20	200	60	400	120
Chloromethane	10	2	40	10	100	20	200	60	400	120
Bromomethane	10	2	40	10	100	20	200	60	400	120
Vinyl chloride	10	2	40	10	100	20	200	60	400	120
Chloroethane	10	2	40	10	100	20	200	60	400	120
Trichlorofluoromethane	10	2	40	10	100	20	200	60	400	120
Acetone[1]	10		40	10	100	20	200	60	400	120
Methylene chloride	5	1	20	5	50	10	100	30	200	60
1,1-Dichloroethene	5	1	20	5	50	10	100	30	200	60
1,1-Dichloroethane	5	1	20	5	50	10	100	30	200	60
trans-1,2-Dichloroethene	5	1	20	5	50	10	100	30	200	60
Methyl *tert*-butyl ether (MTBE)	10		40	10	100	20	200	60	400	120
2,2-Dichloropropane	5	1	20	5	50	10	100	30	200	60
cis-1,2-Dichloroethene	5	1	20	5	50	10	100	30	200	60
Chloroform	5	1	20	5	50	10	100	30	200	60
Bromochloromethane	5	1	20	5	50	10	100	30	200	60
1,2-Dichloroethane	5	1	20	5	50	10	100	30	200	60
Dibromomethane	5	1	20	5	50	10	100	30	200	60
2-Butanone[1]	10		40	10	100	20	200	60	400	120
1,1,1-Trichloroethane	5	1	20	5	50	10	100	30	200	60
Carbon tetrachloride	5	1	20	5	50	10	100	30	200	60
Bromodichloromethane	5	1	20	5	50	10	100	30	200	60
1,2-Dichloropropane	5	1	20	5	50	10	100	30	200	60
cis-1,3-Dichloropropene	5	1	20	5	50	10	100	30	200	60
Trichloroethene	5	1	20	5	50	10	100	30	200	60

Table A-4

Drinking water list Standard: Calibration Levels

Compound	Level 1		Level 2		Level 3		Level 4		Level 5	
Dibromochloromethane	5	1	20	5	50	10	100	30	200	60
1,2-Dibromoethane	5	1	20	5	50	10	100	30	200	60
1,2,3-Trichloropropane	5	1	20	5	50	10	100	30	200	60
1,1,2-Trichloroethane	5	1	20	5	50	10	100	30	200	60
Benzene	5	1	20	5	50	10	100	30	200	60
trans-1,3-Dichloropropene	5	1	20	5	50	10	100	30	200	60
Bromoform	5	1	20	5	50	10	100	30	200	60
4-Methyl-2-pentanone[1]	10		40	10	100	20	200	60	400	120
2-Hexanone[1]	10		40	10	100	20	200	60	400	120
Tetrachloroethene	5	1	20	5	50	10	100	30	200	60
Toluene	5	1	20	5	50	10	100	30	200	60
1,1,2,2-Tetrachloroethane	5	1	20	5	50	10	100	30	200	60
Chlorobenzene	5	1	20	5	50	10	100	30	200	60
Ethylbenzene	5	1	20	5	50	10	100	30	200	60
Styrene	5	1	20	5	50	10	100	30	200	60
m and *p* Xylenes	5	1	20	5	50	10	100	30	200	60
o-xylene	5	1	20	5	50	10	100	30	200	60
Isopropylbenzene	5	1	20	5	50	10	100	30	200	60
Bromobenzene	5	1	20	5	50	10	100	30	200	60
n-Propylbenzene	5	1	20	5	50	10	100	30	200	60
2-Chlorotoluene	5	1	20	5	50	10	100	30	200	60
4-Chlorotoluene	5	1	20	5	50	10	100	30	200	60
1,3,5-Trimethylbenzene	5	1	20	5	50	10	100	30	200	60
tert-Butylbenzene	5	1	20	5	50	10	100	30	200	60
1,2,4-Trimethylbenzene	5	1	20	5	50	10	100	30	200	60
sec-butylbenzene	5	1	20	5	50	10	100	30	200	60
1,3-Dichlorobenzene	5	1	20	5	50	10	100	30	200	60

Table A-4

Drinking water list Standard: Calibration Levels

Compound	Level 1		Level 2		Level 3		Level 4		Level 5	
1,4-Dichlorobenzene	5	1	20	5	50	10	100	30	200	60
1,2-Dichlorobenzene	5	1	20	5	50	10	100	30	200	60
4-Isopropyltoluene	5	1	20	5	50	10	100	30	200	60
n-Butylbenzene	5	1	20	5	50	10	100	30	200	60
1,2-Dibromo-3-chloropropane	5	1	20	5	50	10	100	30	200	60
1,2,4-Trichlorobenzene	5	1	20	5	50	10	100	30	200	60
Napthalene	5	1	20	5	50	10	100	30	200	60
Hexachlorobutadiene	5	1	20	5	50	10	100	30	200	60
1,2,3-Trichlorobenzene	5	1	20	5	50	10	100	30	200	60

† Not included in the Standard test, but included in the standard as a frequently requested add-on.

23. APPENDIX B

This Appendix B lists modifications to the main body of the SOP that are necessary for analysis of water by method 624.

24. MODIFICATIONS REQUIRED FOR ANALYSIS BY EPA 624

24.1. Method 624 is required for demonstration of compliance with NPDES wastewater discharge permits. This method can be applied only to aqueous matrices. The standard analyte list and reporting limits are listed in Table B-1.

24.2. The tune period for this method is defined as 24 hours.

24.3. The initial calibration curve for this method requires at least three points.

24.4. Sample concentrations are calculated using the average RRF from the initial calibration curve.

24.5. Each target analyte is assigned to the closest eluting internal standard.

24.6. *Initial demonstration of Proficiency*

24.6.1. The spiking level for the four replicate initial demonstration of proficiency is 20 µg/L. The acceptance criteria are listed in Table B-2

24.7. Initial calibration curve requirements:

24.7.1. Target compounds must have RSD ≤ 35%.

24.7.2. If this requirement can not be met, a regression curve must be constructed for the non-compliant compounds. There is no correlation coefficient requirement for the regression curve.

24.8. Continuing calibration verification requirements:

24.8.1. The continuing calibration standard is run at 20 µg/L. The acceptance criteria are listed in Table B-2.

24.9. Matrix Spike and LCS requirements

24.9.1. The matrix spike and LCS are spiked at 20 µg/L. A matrix spike duplicate is not necessary for this method. The recovery limits for matrix spike and LCS recovery are listed in Table B-2.

24.10. Method clarifications, modifications and additions

24.10.1. Section 5.2.2 of the source method describes the trap packing materials as Tenax GC, Methyl silicone, silica gel and coconut charcoal. STL routinely employs the Supelco VOCARB 3000, which consists of Carbopack B and Carboxen 1000 and 1001.

24.10.2. Section 5.3.2 of the source method describes a packed analytical column. STL routinely employs capillary columns when performing this method.

24.10.3. The source method provides a suggested list of compounds for internal and surrogate standards. STL uses the following two compounds which are not on the table: Chlorobenzene-d_5 (internal standard) and Toluene-d_8 (surrogate).

Table B-1.
Method 624 Analytes and Reporting Limits

Analytes	µg/L
Benzene	5
Bromodichloromethane	5
Bromoform	5
Bromomethane	5
Carbon tetrachloride	5
Chlorobenzene	5
Chloroethane	5
2-Chloroethyl vinyl ether	5
Chloroform	5
Chloromethane	5
Dibromochloromethane	5
1,2-Dichlorobenzene	5
1,3-Dichlorobenzene	5
1,4-Dichlorobenzene	5
1,1-Dichloroethane	5
1,2-Dichloroethane	5
1,1-Dichloroethene	5
Trans-1,2-Dichloroethene	5
1,2-Dichloropropane	5
cis-1,3-Dichloropropene	5
Trans-1,3-Dichloropropene	5
Ethylbenzene	5
Methylene chloride	5
1,1,2,2-Tetrachloroethane	5
Tetrachloroethene	5
Toluene	5
1,1,1-Trichloroethane	5
1,1,2-Trichloroethane	5
Trichloroethene	5
Trichlorofluoromethane	5
Vinyl chloride	5

Table B-2.
Method 624 QC Acceptance Criteria

Analytes	Daily QC check acceptance criteria (20µg/L spike)	Mean recovery, 4 replicate initial demonstration acceptance criteria (20µg/L spike)	Standard deviation, 4 replicate initial demonstration acceptance criteria (20µg/L spike)	Matrix spike and LCS acceptance criteria (% recovery)
Benzene	12.8-27.2	15.2-26.0	6.9	37-151
Bromodichloromethane	13.1-26.9	10.1-28.0	6.4	35-155
Bromoform	14.2-25.8	11.4-31.1	5.4	45-169
Bromomethane	2.8-37.2	D-41.2	17.9	D-242
Carbon tetrachloride	14.6-25.4	17.2-23.5	5.2	70-140
Chlorobenzene	13.2-26.8	16.4-27.4	6.3	37-160
Chloroethane	7.6-32.4	8.4-40.4	11.4	14-230
2-Chloroethyl vinyl ether	D-44.8	D-50.4	25.9	D-305
Chloroform	13.5-26.5	13.7-24.2	6.1	51-138
Chloromethane	D-40.8	D-45.9	19.8	D-273
Dibromochloromethane	13.5-26.5	13.8-26.6	6.1	53-149
1,2-Dichlorobenzene	12.6-27.4	11.8-34.7	7.1	18-190
1,3-Dichlorobenzene	14.6-25.4	17.0-28.8	5.5	59-156
1,4-Dichlorobenzene	12.6-27.4	11.8-34.7	7.1	18-190
1,1-Dichloroethane	14.5-25.5	14.2-28.5	5.1	59-155
1,2-Dichloroethane	13.6-26.4	14.3-27.4	6.0	49-155
1,1-Dichloroethene	10.1-29.9	3.7-42.3	9.1	D-234
trans-1,2-Dichloroethene	13.9-26.1	13.6-28.5	5.7	54-156
1,2-Dichloropropane	6.8-33.2	3.8-36.2	13.8	D-210
cis-1,3-Dichloropropene	4.8-35.2	1.0-39.0	15.8	D-227
trans-1,3-Dichloropropene	10.0-30.0	7.6-32.4	10.4	17-183
Ethylbenzene	11.8-28.2	17.4-26.7	7.5	37-162
Methylene chloride	12.1-27.9	D-41.0	7.4	D-221
1,1,2,2-Tetrachloroethane	12.1-27.9	13.5-27.2	7.4	46-157
Tetrachloroethene	14.7-25.3	17.0-26.6	5.0	64-148
Toluene	14.9-25.1	16.6-26.7	4.8	47-150
1,1,1-Trichloroethane	15.0-25.0	13.7-30.1	4.6	52-162
1,1,2-Trichloroethane	14.2-25.8	14.3-27.1	5.5	52-150
Trichloroethene	13.3-26.7	18.6-27.6	6.6	71-157
Trichlorofluoromethane	9.6-30.4	8.9-31.5	10.0	17-181
Vinyl chloride	0.8-39.2	D-43.5	20.0	D-251

25. APPENDIX C

This appendix lists modifications to the main body of the SOP necessary for analysis of volatile organic compounds by GC/MS Method 8260A.

26. MODIFICATIONS REQUIRED FOR ANALYSIS BY METHOD 8260A

26.1. Initial Calibration Criteria

26.1.1. Any analyte that gives a % RSD of ≤15% may use average response factor for quantitation, otherwise, a 1st or 2nd order calibration curve must be used for quantitation. All compounds must be quantitated from 1st or 2nd order calibration curves if desired.

26.2. Method 8260A recommends that the purge vessel is run through an additional purge cycle after 25 mL sample analysis to remove carryover. Instead, purge vessels are oven baked between analyses or disposable vessels are used one time only.

26.3. Low Level Soil Prep

26.3.1. Refer to section 8.8 and 11.8 of 8260B for soil sample prep by 5030A.

26.4. Medium Level Soil/Sediment and Waste Samples Prep

26.4.1. Sediments/soils and waste that are insoluble in methanol.

26.4.1.1. Gently mix the contents of the sample container with a narrow metal or wood spatula. Weigh 4 g (wet weight) into a tared vial. Use a top-loading balance. Record the weight to 0.1 gram. Do not discard any supernatant liquids. Liquid waste for TCLP analysis must be aliquoted on a volume basis (i.e. pipette 4-mls of sample instead of 4-grams of sample).

26.4.1.2. Quickly add 9 mL of methanol, and 1 mL of surrogate spiking solution to bring the final volume of methanol to 10 mL. For an LCS or MS/MSD sample, add 8 mL of methanol, 1 ml of surrogate spike solution, and 1 mL of matrix spike solution. Cap the vial and vortex to mix thoroughly.

NOTE: Sections 25.3.1.1 and 25.3.1.2 must be performed rapidly and without interruption to avoid the loss of volatile organics.

26.4.2. Liquid wastes that are soluble in methanol and insoluble in water

26.4.2.1. Pipette 2 mL of the sample into a tared vial. Use a top-loading balance. Record the weight to the nearest 0.1 gram. It is not necessary to record the weight for liquid waste for TCLP analysis since the results are reported on a volume basis.

26.4.2.2. Quickly add 7 mL of methanol, then add 1 mL of surrogate spiking solution to bring the final volume to 10 mL. Cap the vial and shake for 2 minutes to mix thoroughly. For a MS/MSD or LCS, 6 mL of methanol, 1 mL of surrogate solution, and 1 mL of matrix spike solution is used.

26.4.3. Fill a 5 mL syringe with 5 mL of reagent water. Add 100 µL (or less if a dilution is required) of methanol extract from the sample preparation in Section 25.3.1 or 25.3.2. If less than 5 µL of the methanol extract is required, then an intermediate dilution is required. Add 10 µL of the 25 µg/L internal standard solution. (Note that the combined internal standard/surrogate standard solution is not used since surrogates have been added previously.) Inject the sample into the purging chamber and proceed with the analysis per Sections 11.4.4 and 11.4.5.

ENTACT
Appendix
D

ATTACHMENT D

SEVERN TRENT LABORATORY SAMPLE VOLUMES

Sample Volumes
STL St. Louis

This table should be used for guidance only. If there is **any** doubt or question, please consult the appropriate group leader. Some analyses may be able to be combined, such as the anions, with no additional volume required. Check with group leader if there are changes in the method or the manner in which the lab performs an analysis.

Analysis	Method	Matrix	*Volume/Container	Preservative	Hold Time
Br	300.0	Water	100 mL / P,G	Cool, 4 deg. C	28 days
Cl	300.0	Water	100 mL / P,G	Cool, 4 deg. C	28 days
F	300.0, 340.2	Water	100 mL / P,G	Cool, 4 deg. C	28 days
Nitrate/Nitrite	353.1	Water	100 mL / P,G	H2SO4 (pH <2), Cool	28 days
Nitrate	300.0	Water	100 mL / P,G	Cool, 4 deg. C	48 hrs
Nitrite	300.0, 353.1	Water	100 mL / P,G	Cool, 4 deg. C	48 hrs
Orthophos	300.0, 365.1	Water	100 mL / P,G	Cool, 4 deg. C	48 hrs
Perchlorate	300.0	Water	100 mL / P,G	Cool, 4 deg. C	28 days
Sulfate (SO4)	300.0	Water	100 mL / P,G	Cool, 4 deg. C	28 days
Sulfite (SO3)	377.1	Water	100 mL / P,G	Cool, 4 deg. C	24 hrs
Sulfide (S)	9030, 376.1	Water	500 mL / P,G	NaOH,Zn Ac. (pH >9), Cool	7 days
React S	9030	Water	100 mL / P,G	NaOH,Zn Ac. (pH >9), Cool	7 days
React CN	9010	Water	100 mL / P,G	NaOH (pH >12), Cool	14 days
)	9010, 335.2	Water	100 mL / P,G	NaOH (pH >12), Cool	14 days
Cn	CLP	Water	100 mL / P,G	NaOH (pH >12), Cool	12 days**
pH	9040, 150.1	Water	50 mL / P,G	Cool, 4 deg. C	ASAP
BOD	405.1	Water	1000 mL / P,G	Cool, 4 deg. C	48 hrs
CBOD	5210B	Water	1000 mL / P,G	Cool, 4 deg. C	48 hrs
COD	410.4	Water	100 mL / P,G	H2SO4 (pH ,2), Cool	28 days
CO3	310.1	Water	100 mL / P,G	Cool, 4 deg. C	14 days
HCO3	310.1	Water	100 mL / P,G	Cool, 4 deg. C	14 days
Conductivity	120.1, 9050	Water	100 mL / P,G	Cool, 4 deg. C	28 days
Flashpoint	1010	Water	100 mL / P,G	Cool, 4 deg. C	180 days
Hardness	130.2	Water	100 mL / P,G	HNO3 (pH <2)	180 days
Ammonia	350.1	Water	100 mL / P,G	H2SO4 (pH <2), Cool	28 days
Oil & Grease	9070, 413.1	Water	1000 mL / G	HCl (pH <2), Cool	28 days
Phenols	9066, 420.2	Water	100 mL / G	H2SO4 (pH <2), Cool	28 days
Phosphate	365.1	Water	100 mL / P,G	H2SO4 (pH <2), Cool	28 days
React S	9030	Water	10 mL / P,G	Cool, 4 deg. C	None
React CN	9010	Water	10 mL / P,G	Cool, 4 deg. C	None
Settleable Solids		Water	1000 mL / P,G	Cool, 4 deg. C	48 hrs
Surfactants		Water	1000 mL / P,G	Cool, 4 deg. C	48 hrs
Total Solids	160.3	Water	100 mL / P,G	Cool, 4 deg. C	7 days
TSS	160.2	Water	100 mL / P,G	Cool, 4 deg. C	7 days
TDS	160.1	Water	100 mL / P,G	Cool, 4 deg. C	7 days
Turbidity	180.1	Water	100 mL / P,G	Cool, 4 deg. C	48 hrs
`C	415.1	Water	100 mL / P,G	H2SO4 (pH <2), Cool	28 days
JC	9060	Water	100 mL / P,G	H2SO4 (pH <2), Cool	28 days
TOX	450.1	Water	500 mL / G	H2SO4 (pH <2), Cool	28 days

Sample Volumes
STL St. Louis

TOX	9020	Water	500 mL / G	H2SO4 (pH <2), Cool	28 days
Hexachrome	7196A	Water	100 mL / P,G	Cool, 4 deg. C	24 hrs
Br	300.0	Soil	5 g./ P,G	Cool, 4 deg. C	28 days
Cl	300.0	Soil	5 g./ P,G	Cool, 4 deg. C	28 days
Fl	300.0, 340.2	Soil	5 g./ P,G	Cool, 4 deg. C	28 days
Nitrate	353.1	Soil	5 g./ P,G	Cool, 4 deg. C	28 days
Nitrate	300.0	Soil	5 g./ P,G	Cool, 4 deg. C	48 hrs
Nitrite	300.0, 353.1	Soil	5 g./ P,G	Cool, 4 deg. C	48 hrs
Orthophos	300.0, 365.1	Soil	5 g./ P,G	Cool, 4 deg. C	48 hrs
Sulfate (SO4)	300.0	Soil	5 g./ P,G	Cool, 4 deg. C	28 days
Perchlorate	300.0	Soil	5 g./ P,G	Cool, 4 deg. C	28 days
Sulfite (SO3)	377.1	Soil	20 g./ P,G	Cool, 4 deg. C	24 hrs
Sulfide (S)	9030, 376.1	Soil	20 g./ P,G	Cool, 4 deg. C	7 days
React S	9030	Soil	50 g./ P,G	Cool, 4 deg. C	None
React CN	9010	Soil	50 g./ P,G	Cool, 4 deg. C	None
CN	9010, 335.2	Soil	5 g./ P,G	Cool, 4 deg. C	14 days
CN	CLP	Soil	5 g./ P,G	Cool, 4 deg. C	12 days**
pH	9045	Soil	20 g./ P,G	Cool, 4 deg. C	14 days
CO3/HCO3	310.1	Soil	20 g./ P,G	Cool, 4 deg. C	14 days
Conductivity	120.1, 9050	Soil	20 g./ P,G	Cool, 4 deg. C	28 days
Flashpoint	1010	Soil	100 g./ P,G	Cool, 4 deg. C	180 days
Ammonia	350.1	Soil	5 g./ P,G	Cool, 4 deg. C	28 days
Oil & Grease	9071	Soil	20 g./ G	Cool, 4 deg. C	28 days
Paint Filter	9095	Soil	100 g./ P,G	Cool, 4 deg. C	28 days
enols	9066, 420.2	Soil	5 g./ G	Cool, 4 deg. C	28 days
nosphate	365.1	Soil	5 g./ P,G	Cool, 4 deg. C	28 days
TOC	415.1	Soil	25 g./ P,G	Cool, 4 deg. C	28 days
TOC	9060	Soil	25 g./ P,G	Cool, 4 deg. C	28 days
TOX	450.1	Soil	5 g./ G	Cool, 4 deg. C	28 days
TOX	9020	Soil	5 g./ G	Cool, 4 deg. C	28 days
TPH	418.1	Soil	50 g./ G	Cool, 4 deg. C	None
Hexachrome	7196A	Soil	20 g./ P,G	Cool, 4 deg. C	24 hrs
BTEX	8020	Water	3 X 40 mL / G(ZHead)	HCl (pH <2),Cool, 4 deg. C	14 days***
BTEX	8240	Water	3 X 40 mL / G(ZHead)	HCl (pH <2), Cool, 4 deg. C	14 days***
Dioxin	8280, Region 7	Water	1 L / G	Cool, 4 deg. C	30 days
Dioxin	613	Water	1 L / G	Cool, 4 deg. C	7 days
Explosives	8000 GC	Water	100mL / G	Cool, 4 deg. C	7 days
Herbicides	8150	Water	1 L / G	Cool, 4 deg. C	7 days
Metals	6010	Water	1 L / P,G	HNO3 (pH <2)	180 days
Mercury	7470	Water	included in metals/ P	HNO3 (pH <2)	13 days
Mercury	7470	Water	included in metals/ G	HNO3 (pH <2)	38 days
Pest/PCB's	608, 8080	Water	1 L / G	Cool, 4 deg. C	7 days
Semivolatiles	625, 8270	Water	1 L / G	Cool, 4 deg. C	7 days
TPH, Gasoline	8015	Water	3 X 40 mL / G(ZHead)	HCl (pH <2), Cool, 4 deg. C	14 days
TPH, Diesel	8015	Water	1 L / G	HCl (pH <2), Cool, 4 deg. C	7 days
TPH, Diesel	OA2	Water	1 L / G	4 deg. C	7 days
Volatiles	624, 8260	Water	3 X 40 mL / G(ZHead)	HCl (pH <2), Cool, 4 deg.C	14 days***
olatiles	8260(5mL purge)	Water	3 X 40 mL / G(ZHead)	HCl (pH <2), Cool, 4 deg.C	14 days***
atiles	8260(25mL purge)	Water	5 X 40 mL / G(ZHead)	HCl (pH <2), Cool, 4 deg.C	14 days***
Metals	CLP **	Water	1 L / P,G	HNO3 (pH <2)	180 days**

Sample Volumes
STL St. Louis

Mercury	CLP **	Water	included in metals	HNO3 (pH <2)	26 days**
Pest/PCB's	CLP **	Water	1 L / G	Cool, 4 deg. C	5 days**
Semivolatiles	CLP **	Water	1 L / G	Cool, 4 deg. C	5 days**
Volatiles	CLP **	Water	3 X 40 mL / G	HCl (pH <2), Cool, 4 deg.C	10 days**
BTEX	8021-OA1	Soil	120 mL / G (ZHead)	Cool, 4 deg. C	14 days
BTEX	8260	Soil	120 mL / G (ZHead)	Cool, 4 deg. C	14 days
Dioxin	8280/Region 7	Soil	120 mL / G	Cool, 4 deg. C	30 days
Explosives	8000 GC	Soil	120 mL / G	Cool, 4 deg. C	14 days
Herbicides	8150	Soil	250 mL / G	Cool, 4 deg. C	14 days
Metals	6010	Soil	120 mL / G	Cool, 4 deg. C	180 days
Mercury	7470	Soil	included in metals	Cool, 4 deg. C	28 days
Pest/PCB's	8080	Soil	120 mL / G	Cool, 4 deg. C	14 days
Semivolatiles	8270	Soil	120 mL / G	Cool, 4 deg. C	14 days
TPH, Gasoline	8015-OA1	Soil	120 mL / G (ZHead)	Cool, 4 deg. C	14 days
TPH, Diesel	8015-OA2	Soil	120 mL / G	Cool, 4 deg. C	14 days
Volatiles	8260	Soil	120 mL / G (ZHead)	Cool, 4 deg. C	14 days
Volatiles	8260	Soil	Encore Sampler x 2	Cool, 4 deg. C	48 hrs
Metals	CLP **	Soil	120 mL / G	Cool, 4 deg. C	180 days**
Mercury	CLP **	Soil	included in metals	Cool, 4 deg. C	26 days**
Pest/PCB's	CLP **	Soil	120 mL / G	Cool, 4 deg. C	10 days**
Semivolatiles	CLP **	Soil	120 mL / G	Cool, 4 deg. C	10 days**
Volatiles	CLP **	Soil	120 mL / G	Cool, 4 deg. C	10 days**
BTEX	8020	Soil	5 g / G	Cool, 4 deg. C	14 days
BTEX	8240	Soil	5 g / G	Cool, 4 deg. C	14 days
Dioxin	8280/Region 7	Soil	10 g / G	Cool, 4 deg. C	30 days
Explosives	8330 GC/ HPLC	Soil	5 g / G	Cool, 4 deg. C	14 days
Herbicides	8150	Soil	50 g / G	Cool, 4 deg. C	14 days
Metals	6010	Soil	1 g / G	Cool, 4 deg. C	180 days
Mercury	7470	Soil	0.2 g / G	Cool, 4 deg. C	28 days
Pest/PCB's	8080	Soil	30 g / G	Cool, 4 deg. C	14 days
Semivolatiles	8270	Soil	30 g / G	Cool, 4 deg. C	14 days
TPH, Gasoline	8015-OA1	Soil	5 g / G	Cool, 4 deg. C	14 days
TPH, Diesel	8015-OA2	Soil	20 g / G	Cool, 4 deg. C	14 days
Volatiles	8240, 8260	Soil	5 g / G	Cool, 4 deg. C	14 days
Metals	CLP **	Soil	1 g / G	Cool, 4 deg. C	180 days**
Mercury	CLP **	Soil	0.2 g / G	Cool, 4 deg. C	26 days**
Pest/PCB's	CLP **	Soil	30 g / G	Cool, 4 deg. C	10 days**
Semivolatiles	CLP **	Soil	30 g / G	Cool, 4 deg. C	10 days**
Volatiles	CLP **	Soil	5g / G	Cool, 4 deg. C	10 days**
TCLP ZHEVoa	1311/ 8240	Solid	2 X 120 mL /G(ZHead)	Cool, 4 deg. C	14 days
TCLP Semivoa	1311/ 8270	Solid	250 grams / G	Cool, 4 deg. C	14 days
TCLP Pest	1311/ 8080	Solid	250 grams / G	Cool, 4 deg. C	14 days
TCLP Herb	1311/ 8150	Solid	250 grams / G	Cool, 4 deg. C	14 days
TCLP Metals	1311/ 6010/ 7470	Solid	250 grams / G	Cool, 4 deg. C	28 days
CWET and TTLC are the same containers and preservatives as TCLP.					

Sample Volumes
STL St. Louis

Full TCLP Ext:					
CLP ZHEVoa	1311/ 8240	Solid	2 X 120 mL /G(ZHead)	Cool, 4 deg. C	14 days
Full Extraction	1311	Solid	500 grams / G	Cool, 4 deg. C	14 days
		(100%) *			
TCLP ZHEVoa	1311/ 8240	Liquid	3 X 40 mL/G(ZHead)	Cool, 4 deg. C	14 days
TCLP Semivoa	1311/ 8270	Liquid	1 Liter / G	Cool, 4 deg. C	14 days
TCLP Pest	1311/ 8080	Liquid	1 Liter / G	Cool, 4 deg. C	14 days
TCLP Herb	1311/ 8150	Liquid	1 Liter / G	Cool, 4 deg. C	14 days
TCLP Metals	1311/ 6010/ 7470	Liquid	1 Liter / G	Cool, 4 deg. C	180 days/ Hg=28 days
Full TCLP Ext:					
TCLP ZHEVoa	1311/ 8240	Liquid	3 X 40 mL/G(ZHead)	Cool, 4 deg. C	14 days
Full Extraction	1311	Liquid	2 Liters / G	Cool, 4 deg. C	14 days
* Notes:					
1. Clients are required to do the phase separation when a liquid sample is in multiple phases.					
2. When a liquid sample has a % solid content of less than 25 % of the volume, more sample volume will be required to provide an adequate amount of solids for extraction.					
3. For samples requiring Matrix QC, 3 times the volume is required.					

Analysis	Method	Matrix	*Volume/Container	Preservative	Std MDA pCi/L
Gr Alpha/Beta	900.0,9310,7110	Water	200 mL / P,G	HNO3 (pH <2)	** 3 / 4
Gross Alpha	EERF 00.02	Water	500 mL / P,G	HNO3 (pH <2)	3
Tritium	906.0 (distilled)	Water	120 mL / G	None	500
Isotopi Gamma	901, HASL 300	Water	1 L / P,G	HNO3 (pH <2)	20 @ Cs 137
Ra 226 / 228	903.0 / 904.0	Water	1 L / P,G	HNO3 (pH <2)	1 / 1
Sr 89 / 90	Std Mtd 7500-Sr	Water	1 L / P,G	HNO3 (pH <2)	3 / 3
Isotopic U	NAS-NS-3050	Water	1 L / P,G	HNO3 (pH <2)	1
Isotopic Am	NAS-NS-3006	Water	1 L / P,G	HNO3 (pH <2)	1
Isotopic Pu	NAS-NS-3058	Water	1 L / P,G	HN03 (pH <2)	1
Isotopi Am/Pu	see above	Water	1 L / P,G	HNO3 (pH <2)	1 / 1
Isotopic Th	NAS-NS-3004	Water	1 L / P,G	HNO3 (pH <2)	1
Technetium 99	HASL 300 E-TC-01	Water	1 L / P,G	HNO3 (pH <2)	10
C 14	EERF C-01	Water	1 L / P,G	None	300
Polonium 210	HASL 300 PO-01	Water	1 L / P,G	HNO3 (pH <2)	1
Lead 210	EERF PB-01	Water	1 L / P,G	HNO3 (pH <2)	5
Total Uranium	ASTM 5174-91	Water	5 mL / P,G	HNO3 (pH <2)	1 or 1 ug/L

Analysis	Method	Matrix	*Volume/Container	Preservative	pCi/g
Gr Alpha/Beta	9310, 7110	Soil	1 g / P,G	None	10 / 10
Tritium	EERF H.01	Soil	100 g / G	None	5
Isotopic Gamma	HASL 300	Soil	650 g / P,G	None	0.2 @ Cs137
226 / 228	HASL 300	Soil	650 g / P,G	None	1 / 1
89 / 90	Std Mtd 7500-Sr	Soil	10 g / P,G	None	3 / 3
Isotopic U	NAS-NS-3050	Soil	10 g / P,G	None	1

Sample Volumes
STL St. Louis

Isotopic Am	NAS-NS-3006	Soil	10 g / P,G	None	1
Isotopic Pu	NAS-NS-3058	Soil	10 g / P,G	None	1
Isotopic Am/Pu	see above	Soil	10 g / P,G	None	1 / 1
Isotopic Am	HASL 300 (Gam)	Soil	650 g / P,G	None	3
Isotopic Th	NAS-NS-3004	Soil	10 g / P,G	None	1
Technetium 99	HASL 300 E-TC-01	Soil	10 g / P,G	None	10
Carbon 14	EERF C-01	Soil	5 g / P,G	None	300
Polonium 210	HASL 300 PO-01	Soil	1 g / P,G	None	1
Lead 210	EERF PB-01	Soil	5 g / P,G	None	5
Total Uranium	ASTM 5174-91	Soil	1 g / P,G	None	5 ug/g
					or 5 pCi/g

Note: 1. For Product Codes and additional description, see STANDARD PRODUCT LIST.

2. Matrix spikes not performed on soils for Isotopic Gamma or Gross Alpha/Beta analyses.

3. Hold time for radiochemistry analyses is 180 days.

* Sample volumes are based on dry weights, volumes need to be increased if soil is wet/moist.

For samples requiring Matrix QC, 3 times the volume is required.

For normal samples, 2 or more times the volume may be required for re-extracts/digestions.

** Gross Alpha MDA is achievable only when solids are less than 500 ppm.

Greyed areas are isotope analyses performed at Quanterra-Richland.

ENTACT
Appendix
E

ATTACHMENT E

CHAIN OF CUSTODY FORM, CUSTODY SEAL AND SAMPLE LABELS

CHAIN OF CUSTODY

ENTACT
1360 N. Wood Dale Rd. Suite A
Wood Dale, Illinois 60191
Ph. 630/616-2100 Fax 630/616-9203

Sampler:_____ Job #:_____

ENTACT Contact:_____ Date:_____

Turnaround Time Requested

24 Hour ☐ 48 Hour ☐ 3 Day ☐ Normal ☐ Other ☐ _____

Sample No.	Matrix	Composite or Grab	Description/Remarks	Preservative	Analysis

Samples Relinquished By: _____ Date

Samples Received By: _____ Date

Samples Relinquished By: _____ Date

Samples Received By: _____ Date

Samples Relinquished By: _____ Date

ANALYSIS

A=_____ F=_____

B=_____ G=_____

C=_____ H=_____

D=_____ I=_____

E=_____ J=_____

Condition of Sample Upon Receipt:

Bottles Intact? Yes / No	Volatiles Free of Headspace? Yes / No	COC Seals Present and Intact? Yes / No

Distribution:

Original - To Customer w/ Final Report
2nd Copy - To Job File
3rd Copy - To Lab

ENTACT
Appendix
F

APPENDIX F

XRF STANDARD OPERATING PROCEDURES

X-RAY FLUORESCENCE (XRF) ANALYSIS OF SOIL STANDARD OPERATING PROCEDURES

FOR

THE OLD AMERICAN ZINC SITE
FAIRMONT CITY, ILLINOIS

PREPARED BY:
ENTACT, Inc.

April, 2002

Table of Contents

I. Principle, Scope and Application

The purpose of this standard operating procedure (SOP) is to serve as a guide for the field analysis of soils for metals. The procedures herein are general operating procedures for the Spectrace 9000 XRF Analyzer or equivalent. They contain detailed procedures for calibration, operation and maintenance of the XRF.

X-radiation of sufficient energy will cause all atoms to fluoresce, emitting x-rays of characteristic energy. By analyzing the fluorescent radiation emitted by a sample under excitation, both the identity and the quantity of the elements present in the sample can be determined.

II. Parameters to Be Measured

A. Lead is the contaminant of concern at this site and will be the only metal measured and reported by the XRF.

III. Range Of Measurement

A. The range of measurements for lead is 50 ppm through 300,000 ppm.

IV. Detection Limit

A. The detection limit is variable with each analysis. The detection limit for each analysis is three times the XRF calculated standard deviation.

Example:
1. The XRF calculated standard deviation is 5 ppm.
2. 5 X 3 = 15
3. The detection limit is 15 ppm.

V. Sample Matrix

A. The SOP is applicable to both in-situ and ex-situ soils and waste.

VI. Interference and Corrective Actions

A. Lead - Arsenic Interference

1. Interference

Due to the close proximity of the spectra for lead and arsenic, arsenic levels may be masked when the arsenic levels are less than 10% that of lead.

2. Corrective Action

TN Technologies has developed an additional software package for the Spectrace 9000 that will allow the XRF to detect arsenic when levels are as low as 5% that of lead.

B. Moisture

1. Interference
   High moisture content (approximately 20% moisture) of muds and sludges can cause erroneous results.

2. Corrective Action
   Soils containing high moisture content should be dried prior to analysis.

C. Matrix Effects

1. Interference
   Physical characteristics such as particle size and homogeneity can affect the accuracy of the analysis.

2. Corrective Action
   Whenever a new matrix is encountered a sample should be analyzed by both XRF and the laboratory analysis to ensure the XRF accurately analyzes the constituents in the matrix.

D. Placement

1. Interference
   If the XRF probe is not placed on a flat uniform soil location errors can result from the distance between the probe and the soil.

2. Corrective Action
   Ensure with each measurement that the probe window is placed flat against a uniform flat surface.

VII. Safety Precautions and Emergency Procedures

The State of Illinois Department of Health, Bureau of Radiation Protection will be properly notified prior to bringing the XRF instrument to the site.

A. Radiation Levels

According to the Spectrace 9000 user manual, the radiation exposure rate due to the XRF sources with the shutters closed is <0.1 mR/h. In addition, while the shutters are open, the exposure ate remains low provided a sample is completely covering the probe window. The XRF should never be run without a sample over the probe window.

B. Shipment

Under U.S. DOT regulations (49 CFR, 173.422) and International Air Transport Association (IATA), the XRF unit is classified as "Radioactive material, excepted package, instruments, UN2910." As such, the device can be transported by any mode

including air, land or sea. It is eligible to be transported in the baggage compartment of a passenger-carrying aircraft. The device is excepted from all specification packaging, marking and labeling. The bill of lading should, however, contain the words: "Radioactive material, excepted package, instruments, UN2910."

C. Storage

ENTACT's XRF units are generally licensed to either the Dallas, Texas or Chicago, Illinois office. The units are to be permanently stored in the office, whether Dallas or Chicago, to which it is licensed. The units can be transported to and temporarily (less than 30 days) stored in another state without the state being notified. If it is going to be transported to and stored in another state for longer than 30 days, that state must be contacted to determine the process involved with registering the XRF in that state.

D. Emergency Procedures

1. Secure the area around the incident. Keep unauthorized persons away. Alert people in vicinity of radioactive material and possible hazards.

2. DO NOT LEAVE THE SITE. Send a helper to notify the following persons:

   Radiation Safety Officer (RSO):To be assigned
   Work Phone:To be assigned
   Pager: To be assigned
   Home Phone: To be assigned
   Local Fire and Police Departments 911

3. The Radiation Safety Officer will provide appropriate notification to:

   Illinois Department of Nuclear Safety: (630) 293-8252
   TN Technologies Inc.: (512) 388-9285 or (512) 388-9287

4. The RSO or alternate should inform emergency workers of the potential for existence of a radiation hazard; should help keep the area secure; and should explain to emergency personnel the location of the radioactive device and the extent of the possible hazard. In no case should the response personnel leave the site until qualified experts arrive, unless the worker is seriously injured or incapacitated, and must be removed from the site by emergency personnel.

   If the RSO cannot be reached, notify Jonathon Patlak.

   Work Number: (630) 616-2100
   Home Number: (847) 412-1917

VIII. Sample Size, Collection, Preservation and Handling

A. The sample size, collection and handling requirements for samples undergoing XRF analysis are determined on a site-specific basis. These are to be addressed in the site work plan and quality control plan. The exact requirements will vary depending on the use of the XRF on the site. No preservation is required for soils that are to be analyzed for metals.

IX. Apparatus and Materials

A. Probe

The probe consists of a sealed aluminum enclosure containing a high-resolution mercuric iodide detector and three radioisotope x-ray excitation sources. The probe aperture window, through which the analysis is performed, is sealed with thin replaceable film. The probe also contains a pre-amplifier and bias supply for the detector and a mechanism to move the radioisotope sources from their shielded location during an analysis.

B. Electronics Unit

The electronics unit provides data acquisition, processing, and display capabilities. The computer includes a math coprocessor for fast calculation of results. Sufficient memory is available to store up to 300 sets of analysis and 120 spectra. An RS-232 port allows stored data to be transferred to another computer. The graphics display allows direct viewing and qualitative analysis of the x-ray spectra. The replaceable and rechargeable internal battery provides for field portable operation.

C. Additional Parts and Accessories

Additional parts and accessories include: the interconnecting able, battery chargers, RS-232C interface cable, carrying case, carrying bag, spare battery, analysis stand, Teflon bank and metal standards.

X. Routine Preventative Maintenance

-ENTACT identifies each XRF result with a unique identification number which all-routine preventative maintenance to be accomplished as follows:

A. Standardization

The XRF must be standardized by technicians at TN Technologies on an annual basis.

B. Leak Tests

The XRF must be leak tested by technicians at TN Technologies every six months.

C. Source Change

The sources on the XRF must be changed by technicians at TN Technologies according to the following schedule:

Cd-109	2.5 years
Fe-55	5 years
Am 241	never

D. Film change

The film covering the aperture window needs to be changed when it becomes damaged.

XI. Calibration Standards

A. Site Specific Standards

1. Preparation

a. Collect three soil samples from the site in which XRF analysis will be performed. Use the XRF to guide the collection process. Try to collect samples that vary over the range of total lead levels characteristic of site concentrations.

b. Transport the samples to the lab and instruct the analyst to perform the following in the order listed for each sample:

-Dry the samples
-Grind the samples into a fine powder, removing any rocks or debris
-Homogenize each individual sample
-Split each sample. Return one half to ENTACT for use as the standard. Analyze the other half five times for total lead. The lab should then average the results giving a "certified value".

c. Prepare the site-specific standards using the returned portions of the samples. Place the soil into the XRF sample cups, cover with film and seal. The total lead value of the standard is the average of the five laboratory total lead values.

d. Use three of the prepared standards to calibrate the XRF. Select one standard below the clean-up criteria, one standard above the clean-up criteria but below the treatment correlation value, and one standard above the treatment correlation value.

2. Storage

a. The standards must be stored in a manner that will prevent damage to the film.

b. The shelf life of the site-specific standards is 6 months. Upon expiration of these standards, the standard value should be re-certified by submitting additional sample to the laboratory for re-analysis.

B. Teflon

1. Storage

a. The Teflon standard must be stored in a manner that will prevent damage and contamination.

b. These standards have an unlimited shelf life.

C. Pure Metal Standards

1. Storage

a. The five pure metal standards (lead, iron, tin, titanium and zinc) standards must be stored in a manner that will prevent damage and contamination.

b. These standards have an unlimited shelf life.

XII. Calibration Procedures

-The following procedures should be performed at the beginning of each days analysis. In addition one site-specific standard to be analyzed for every twenty sample locations analyzed. Finally, at the end of the day all three site-specific standards should be re-analyzed.

A. Instrument Set-up

1. Place the electronics portion of the XRF on a flat surface, adjusting the handle to be used as a stand.

2. Connect the Electronics portion to the probe using the interconnecting cable.

a. When inserting the cable into the probe and electronics portion, pull back metal cover on end of the cable, align the red dot on the cable with the grove on the insertion point and finally gently insert the cable until you hear a soft "click".

3. Remove the safety cover from the probe.

4. Place the probe on the lab stand base.

5. Secure the shield cup to the top of the probe.

B. Turn on procedures

1. Turn on the unit.

a. Press the "On" button.

b. You will then receive the prompt, "Is 0:00:00 the correct time?" If it is the correct time, press "yes" (the number 1 button). If it is not the correct date, press "no" (the number 2 button). The XRF will then instruct you on how to reset the time.

c. You will then receive the prompt, "Is 0:00:00 the correct date?". If it is the correct date, press "yes" (the number 1 button). If it is not the correct date, press "no" (the number 2 button). The XRF will then instruct you on how to reset the date.

d. Allow the XRF to warm-up for at least 10 minutes.

C. Calibration

1. You are now at the main menu. Select measure (press the number 1 button).

2. You now need to modify the scanning time to allow 50 seconds per source to scan the iron standard.

a. Select "modify" (press the number 1 button).

b. Select the "Mod" (press the number 3 button).

c. Enter 50 and press the Cont/Pause button.

d. Select "Down" (press the number 2 button).

e. Select "Mod" (press the number 3 button).

f. Enter 50 and press the Cont/Pause button).

g. Select "Down" (press the number 2 button).

h. Select "Mod" (press the number 3 button).

i. Enter 50 and press the Cont/Pause button.

j. Select "Exit" (press the number 6 button).

3. You are now ready to analyze the iron (FE) standard.

a. Place the iron standard over the source window.

b. Close the shield cup lid.

c. Press the Cont/Pause button.

d. You will now see the label screen.

e. Select the column with "F" in it (press the number 2 button).

f. Select "F" (press the number 6 button).

g. Select the column with "E" in it (press the number 2 button).

h. Select "E" (press the number 5 button).

i. Press the Cont/Pause button.

j. Select "Opts" (press the number 5 button).

k. Select "See raw data" (press the number 5 button).

l. Select "Cd109 33" (press the number 1 button).

m. Select "Intensities" (press the number 6 button).

n. Select "Down" (press the number 2 button) until you can read the value for iron (FE). This value should be between 0.98 and 1.02. If it is not, perform an energy calibration. The procedures for an energy calibration are discussed in Section D of this section.

o. Select "Quit" (press the number 6 button).

p. Select "Quit" (press the number 7 button).

q. Select "EXIT" (press the number 0 button).

4. You now need to modify the scanning time for all three sources to measure the Teflon standard.

a. Select "Measure" (press the number 1 button).

b. Select "modify" (press the number 1 button).

c. Select "Mod" (press the number 3 button).

d. Enter 200 and press the Cont/Pause button.

e. Select "Down" (press the number 2 button).

f. Select "Mod" (press the number 3 button).

g. Enter 200 and press the Cont/Pause button.

h. Select "Down" (press the number 2 button).

i. Select "Mod" (press the number 3 button).

j. Enter 200 and press the Cont/Pause button.

k. Select "Exit" (press the number 6 button).

5. You are now ready to analyze the Teflon standard.

a. Place the Teflon standard over the source window.

b. Close the shield cup lid.

c. Press the Cont/Pause button.

d. You will now see the label screen.

e. Select the column with "T" in it (press the number 5 button).

f. Select "T" (press the number 6 button).

g. Select the column with "E" in it (press the number 2 button).

h. Select "E" (press the number 5 button).

i. Select the column with "F" in it (press the number 2 button).

j. Select "F" (press the number 6 button).

k. Select the column with "L" in it (press the number 3 button).

l. Select "L" (press the number 6 button).

m. Select the column with "O" in it (press the number 4 button).

n. Select "O" (press the number 3 button).

o. Select the column with "N" in it (press the number 4 button).

p. Select "N" (press the number 2 button).

q. Press the Cont/Pause button.

r. Press the zero button.

s. Select "Page down" (press the number 2 button).

t. For all results, the result divided by the standard deviation should be less than five (5). If it is not, acquire new background data are discussed in Section E of this section.

6. You now need to modify the scanning times for site specific calibration.

   a. Select "modify" (press the number 1 button).

   b. Select "Mod" (press the number 3 button).

   c. Enter 40 and press the Cont/Pause button.
   d. Select "Down" (press the number 2 button).

   e. Select "Mod" (press the number 3 button).

   f. Enter 10 and press the Cont/Pause button.

   g. Select "Down" (press the number 2 button).

   h. Select "Mod" (press the number 3 button).

   i. Enter 10 and press the Cont/Pause button.

   j. Select "Exit" (press the number 6 button).

7. You are now ready to analyze the site-specific standards.

   a. Place one of the site-specific standards over the source window.

   b. Close the shield cup lid.

   c. Press the Cont/Pause button.

   d. You will now see the label screen.

   e. Select the column with the first letter or number of your standard name (press the appropriate number button).

   f. Continue this process for the entire standard label.

   g. Press the Cont/Pause button.

h. Press the zero button.

i. Select "Page down" (press the number 2 button).

j. Note the value for lead (Pb) or whatever elements for which you are analyzing the samples.

k. Repeat steps c-j for the standard two more times. Each standard should be analyzed in triplicate.

l. The average of the three values found for the standard should be within ± 20% of the known value of the standard. If it is now, perform an energy calibration. The procedures for an energy calibration are discussed in Section D of this section.

m. Repeat steps a-l for all other site-specific standards.

The XRF is now ready to be used.

D. Energy Calibration

1. You are now at the Main menu. Select measure (press the number 1 button).
2. Select "Options" (press the number 5 button).
3. Select "Energy calibration" (press the number 1 button).
4. The XRF will then say "Measure Safety Cover".
5. Put the safety cover on the probe.
6. Select "Proceed" (press the number 1 button).
7. The XRF will return to the analysis screen when the energy calibration is complete.

E. Background Data Acquisition

1. You are now at the Main menu. Select measure (press the number 1 button).
2. Select "Options" (press the number 5 button).
3. Select "Acquire background data" (press the number 2 button).
4. The XRF will then say "Measure Quartz".
5. Put the quartz standard on the probe.

6. Select "Proceed" (press the number 1 button).

7. The XRF will return to the analysis screen complete and give further instructions. Follow these instructions until acquisition is complete.

XIII. Sample Preparation

A. In-situ Samples

1. Clear the soil of all vegetation.
2. Clear the soil of any debris that may puncture the aperture window.
3. Tamp the soil to ensure it is flat and free of voids.

B. Collected Samples

1. Dry the samples in an oven or microwave oven.
2. Grind the samples into a fine powder, removing any large rocks or debris.
3. Homogenize the sample to ensure consistency.
4. Place the soil into an XRF soil cup, cover with film and seal.

XIV. Analytical Measurement

A. Instrument Set-up

1. Place the electronics portion of the XRF on a flat surface, adjusting the handle to be used as a stand.

2. Connect the Electronics portion to the probe using the interconnecting cable.

   a. When inserting the cable into the probe and electronics portion, pull back metal cover on end of the cable, align the red dot on the cable with the grove on the insertion point and finally gently insert the cable until you hear a soft "click".

3. Remove the safety cover from the probe.

B. Turn on procedures

1. Turn on the unit.
   a. Press the "On" button.

   b. You will then receive the prompt, "Is 0:00:00 the correct time?". If it is the correct time, press "yes" (the number 1 button). If it is now the correct time, press "no" (the number 2 button). The XRF will then instruct you on how to reset the time.

   c. You will then receive the prompt, "Is 0:00:00 the correct date?" If it is the correct date, press "yes" (the number 1 button). If it is not the correct date, press "no" (the number 2 button). The XRF will then instruct you on how to reset the date.

d. Allow the XRF to warm-up for at least 10 minutes.

C. Field use

1. You are now at the main menu. Select measure (press the number 1 button).
2. You now may need to modify the scanning time.
    a. Select "modify" (press the number 1 button).
    b. Select "Mod" (press the number 3 button).
    c. Enter 40 and press the Cont/Pause button.
    d. Select "Down" (press the number 2 button).
    e. Select "Mod" (press the number 3 button).
    f. Enter 10 and press the Cont/Pause button.
    g. Select "Down" (press the number 2 button).
    h. Select "Mod" (press the number 3 button).
    i. Enter 10 and press the Cont/Pause button.
    j. Select "Exit" (press the number 6 button).
3. You are now ready for analysis
    a. Place one of the samples over the source window or place the probe on the area to be analyzed making sure the window is not punctured.
    b. Close the shield cup lid if applicable.
    c. Press the Cont/Pause button.
    d. You will now see the label screen.
    e. Select the column with the first letter or number of your sample name (press the appropriate number button).
    f. Continue this process for the entire sample label.
    g. Press the Cont/Pause button.

h. Press the zero button.

i. Select "Page down" (press the number 2 button).

j. Note the value for lead (Pb) or whatever elements for which you are analyzing the samples.

k. Repeat steps c-j for the standard two more times. Each standard should be analyzed in triplicate.

XV. Data Treatment

A. The result at each sample location is the average of three reading taken at the location.

Result = (Reading 1 + Reading 2 + Reading 3) / 3

B. All readings must be greater than three times the XRF calculated standard deviation in order to be considered valid.

Reading > 3 * Standard deviation

If the above level is not achieved increase the scan time until it is achieved.

XVI. Data Deliverables

-The following documents are available to the client upon request:

A. A summary of initial, ongoing and end of analysis calibration results. This should include each reading, the average of the three readings for each sit-specific standard and the percent difference between the result and the laboratory determined value.

B. A logbook detailing the following:

1. Weather conditions
2. Sampler/s
3. Date of analysis
4. Time of each analysis
5. Location of each analysis
6. Sample preparations required
7. Results of each analysis
8. Any problems encountered and corrective actions taken
9. List date of XRF purchase, latest calibration, leak test and source replacement

C. A printout of all results saved on the XRF and downloaded to a PC. This will be downloaded and formatted in EXCEL and will include sample number, date taken and value in ppm.

D. A summary of all QC required. This will be determined on a site specific basis.

XVII. Quality Control Requirements

A. The quality control requirements for the use of the XRF are determined on a site specific basis. These are to be addressed in the site work plan and quality control plan. The exact requirements will vary depending on the use of the XRF on the site. However, all plans should require instrument calibration prior to and after XRF usage.

XVIII. References

A. Spectrace 9000 Analyzer Manual
TN Technologies Inc.
1992, 1993 and 1994

B. Quality Assurance Technical Information Bulletin
US Environmental Protection Agency
Vol. 1, No. 4
May 1991

X-RAY FLUORESCENCE (XRF) ANALYSIS OF SOIL STANDARD OPERATING PROCEDURES

FOR

THE OLD AMERICAN ZINC SITE
FAIRMONT CITY, ILLINOIS

PREPARED BY:
ENTACT, Inc.

April, 2002

Table of Contents

I. Principle, Scope and Application

The purpose of this standard operating procedure (SOP) is to serve as a guide for the field analysis of soils for metals. The procedures herein are general operating procedures for the Spectrace 9000 XRF Analyzer or equivalent. They contain detailed procedures for calibration, operation and maintenance of the XRF.

X-radiation of sufficient energy will cause all atoms to fluoresce, emitting x-rays of characteristic energy. By analyzing the fluorescent radiation emitted by a sample under excitation, both the identity and the quantity of the elements present in the sample can be determined.

II. Parameters to Be Measured

A. Lead is the contaminant of concern at this site and will be the only metal measured and reported by the XRF.

III. Range Of Measurement

A. The range of measurements for lead is 50 ppm through 300,000 ppm.

IV. Detection Limit

A. The detection limit is variable with each analysis. The detection limit for each analysis is three times the XRF calculated standard deviation.

Example:
1. The XRF calculated standard deviation is 5 ppm.
2. 5 X 3 = 15
3. The detection limit is 15 ppm.

V. Sample Matrix

A. The SOP is applicable to both in-situ and ex-situ soils and waste.

VI. Interference and Corrective Actions

A. Lead - Arsenic Interference

1. Interference

Due to the close proximity of the spectra for lead and arsenic, arsenic levels may be masked when the arsenic levels are less than 10% that of lead.

2. Corrective Action

TN Technologies has developed an additional software package for the Spectrace 9000 that will allow the XRF to detect arsenic when levels are as low as 5% that of lead.

B. Moisture

1. Interference
   High moisture content (approximately 20% moisture) of muds and sludges can cause erroneous results.

2. Corrective Action
   Soils containing high moisture content should be dried prior to analysis.

C. Matrix Effects

1. Interference
   Physical characteristics such as particle size and homogeneity can affect the accuracy of the analysis.

2. Corrective Action
   Whenever a new matrix is encountered a sample should be analyzed by both XRF and the laboratory analysis to ensure the XRF accurately analyzes the constituents in the matrix.

D. Placement

1. Interference
   If the XRF probe is not placed on a flat uniform soil location errors can result from the distance between the probe and the soil.

2. Corrective Action
   Ensure with each measurement that the probe window is placed flat against a uniform flat surface.

VII. Safety Precautions and Emergency Procedures

The State of Illinois Department of Health, Bureau of Radiation Protection will be properly notified prior to bringing the XRF instrument to the site.

A. Radiation Levels

According to the Spectrace 9000 user manual, the radiation exposure rate due to the XRF sources with the shutters closed is <0.1 mR/h. In addition, while the shutters are open, the exposure ate remains low provided a sample is completely covering the probe window. The XRF should never be run without a sample over the probe window.

B. Shipment

Under U.S. DOT regulations (49 CFR, 173.422) and International Air Transport Association (IATA), the XRF unit is classified as "Radioactive material, excepted package, instruments, UN2910." As such, the device can be transported by any mode

including air, land or sea. It is eligible to be transported in the baggage compartment of a passenger-carrying aircraft. The device is excepted from all specification packaging, marking and labeling. The bill of lading should, however, contain the words: "Radioactive material, excepted package, instruments, UN2910."

C. Storage

ENTACT's XRF units are generally licensed to either the Dallas, Texas or Chicago, Illinois office. The units are to be permanently stored in the office, whether Dallas or Chicago, to which it is licensed. The units can be transported to and temporarily (less than 30 days) stored in another state without the state being notified. If it is going to be transported to and stored in another state for longer than 30 days, that state must be contacted to determine the process involved with registering the XRF in that state.

D. Emergency Procedures

1. Secure the area around the incident. Keep unauthorized persons away. Alert people in vicinity of radioactive material and possible hazards.

2. DO NOT LEAVE THE SITE. Send a helper to notify the following persons:

   Radiation Safety Officer (RSO):To be assigned
   Work Phone:To be assigned
   Pager: To be assigned
   Home Phone: To be assigned
   Local Fire and Police Departments 911

3. The Radiation Safety Officer will provide appropriate notification to:

   Illinois Department of Nuclear Safety: (630) 293-8252
   TN Technologies Inc.: (512) 388-9285 or (512) 388-9287

4. The RSO or alternate should inform emergency workers of the potential for existence of a radiation hazard; should help keep the area secure; and should explain to emergency personnel the location of the radioactive device and the extent of the possible hazard. In no case should the response personnel leave the site until qualified experts arrive, unless the worker is seriously injured or incapacitated, and must be removed from the site by emergency personnel.

   If the RSO cannot be reached, notify Jonathon Patlak.

   Work Number: (630) 616-2100
   Home Number: (847) 412-1917

VIII. Sample Size, Collection, Preservation and Handling

A. The sample size, collection and handling requirements for samples undergoing XRF analysis are determined on a site-specific basis. These are to be addressed in the site work plan and quality control plan. The exact requirements will vary depending on the use of the XRF on the site. No preservation is required for soils that are to be analyzed for metals.

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B. Electronics Unit

The electronics unit provides data acquisition, processing, and display capabilities. The computer includes a math coprocessor for fast calculation of results. Sufficient memory is available to store up to 300 sets of analysis and 120 spectra. An RS-232 port allows stored data to be transferred to another computer. The graphics display allows direct viewing and qualitative analysis of the x-ray spectra. The replaceable and rechargeable internal battery provides for field portable operation.

C. Additional Parts and Accessories

Additional parts and accessories include: the interconnecting able, battery chargers, RS-232C interface cable, carrying case, carrying bag, spare battery, analysis stand, Teflon bank and metal standards.

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B. Leak Tests

The XRF must be leak tested by technicians at TN Technologies every six months.

C. Source Change

The sources on the XRF must be changed by technicians at TN Technologies according to the following schedule:

Cd-109	2.5 years
Fe-55	5 years
Am 241	never

D. Film change

The film covering the aperture window needs to be changed when it becomes damaged.

XI. Calibration Standards

A. Site Specific Standards

1. Preparation

a. Collect three soil samples from the site in which XRF analysis will be performed. Use the XRF to guide the collection process. Try to collect samples that vary over the range of total lead levels characteristic of site concentrations.

b. Transport the samples to the lab and instruct the analyst to perform the following in the order listed for each sample:

-Dry the samples
-Grind the samples into a fine powder, removing any rocks or debris
-Homogenize each individual sample
-Split each sample. Return one half to ENTACT for use as the standard. Analyze the other half five times for total lead. The lab should then average the results giving a "certified value".

c. Prepare the site-specific standards using the returned portions of the samples. Place the soil into the XRF sample cups, cover with film and seal. The total lead value of the standard is the average of the five laboratory total lead values.

d. Use three of the prepared standards to calibrate the XRF. Select one standard below the clean-up criteria, one standard above the clean-up criteria but below the treatment correlation value, and one standard above the treatment correlation value.

2. Storage

a. The standards must be stored in a manner that will prevent damage to the film.

b. The shelf life of the site-specific standards is 6 months. Upon expiration of these standards, the standard value should be re-certified by submitting additional sample to the laboratory for re-analysis.

B. Teflon

1. Storage

a. The Teflon standard must be stored in a manner that will prevent damage and contamination.

b. These standards have an unlimited shelf life.

C. Pure Metal Standards

1. Storage

a. The five pure metal standards (lead, iron, tin, titanium and zinc) standards must be stored in a manner that will prevent damage and contamination.

b. These standards have an unlimited shelf life.

XII. Calibration Procedures

-The following procedures should be performed at the beginning of each days analysis. In addition one site-specific standard to be analyzed for every twenty sample locations analyzed. Finally, at the end of the day all three site-specific standards should be re-analyzed.

A. Instrument Set-up

1. Place the electronics portion of the XRF on a flat surface, adjusting the handle to be used as a stand.

2. Connect the Electronics portion to the probe using the interconnecting cable.

a. When inserting the cable into the probe and electronics portion, pull back metal cover on end of the cable, align the red dot on the cable with the grove on the insertion point and finally gently insert the cable until you hear a soft "click".

3. Remove the safety cover from the probe.

4. Place the probe on the lab stand base.

5. Secure the shield cup to the top of the probe.

B. Turn on procedures

1. Turn on the unit.

a. Press the "On" button.

b. You will then receive the prompt, "Is 0:00:00 the correct time?" If it is the correct time, press "yes" (the number 1 button). If it is not the correct date, press "no" (the number 2 button). The XRF will then instruct you on how to reset the time.

c. You will then receive the prompt, "Is 0:00:00 the correct date?". If it is the correct date, press "yes" (the number 1 button). If it is not the correct date, press "no" (the number 2 button). The XRF will then instruct you on how to reset the date.

d. Allow the XRF to warm-up for at least 10 minutes.

C. Calibration

1. You are now at the main menu. Select measure (press the number 1 button).

2. You now need to modify the scanning time to allow 50 seconds per source to scan the iron standard.

a. Select "modify" (press the number 1 button).

b. Select the "Mod" (press the number 3 button).

c. Enter 50 and press the Cont/Pause button.

d. Select "Down" (press the number 2 button).

e. Select "Mod" (press the number 3 button).

f. Enter 50 and press the Cont/Pause button).

g. Select "Down" (press the number 2 button).

h. Select "Mod" (press the number 3 button).

i. Enter 50 and press the Cont/Pause button.

j. Select "Exit" (press the number 6 button).

3. You are now ready to analyze the iron (FE) standard.

a. Place the iron standard over the source window.

b. Close the shield cup lid.

c. Press the Cont/Pause button.

d. You will now see the label screen.

e. Select the column with "F" in it (press the number 2 button).

f. Select "F" (press the number 6 button).

g. Select the column with "E" in it (press the number 2 button).

h. Select "E" (press the number 5 button).

i. Press the Cont/Pause button.

j. Select "Opts" (press the number 5 button).

k. Select "See raw data" (press the number 5 button).

l. Select "Cd109 33" (press the number 1 button).

m. Select "Intensities" (press the number 6 button).

n. Select "Down" (press the number 2 button) until you can read the value for iron (FE). This value should be between 0.98 and 1.02. If it is not, perform an energy calibration. The procedures for an energy calibration are discussed in Section D of this section.

o. Select "Quit" (press the number 6 button).

p. Select "Quit" (press the number 7 button).

q. Select "EXIT" (press the number 0 button).

4. You now need to modify the scanning time for all three sources to measure the Teflon standard.

a. Select "Measure" (press the number 1 button).

b. Select "modify" (press the number 1 button).

c. Select "Mod" (press the number 3 button).

d. Enter 200 and press the Cont/Pause button.

e. Select "Down" (press the number 2 button).

f. Select "Mod" (press the number 3 button).

g. Enter 200 and press the Cont/Pause button.

h. Select "Down" (press the number 2 button).

i. Select "Mod" (press the number 3 button).

j. Enter 200 and press the Cont/Pause button.

k. Select "Exit" (press the number 6 button).

5. You are now ready to analyze the Teflon standard.

a. Place the Teflon standard over the source window.

b. Close the shield cup lid.

c. Press the Cont/Pause button.

d. You will now see the label screen.

e. Select the column with "T" in it (press the number 5 button).

f. Select "T" (press the number 6 button).

g. Select the column with "E" in it (press the number 2 button).

h. Select "E" (press the number 5 button).

i. Select the column with "F" in it (press the number 2 button).

j. Select "F" (press the number 6 button).

k. Select the column with "L" in it (press the number 3 button).

l. Select "L" (press the number 6 button).

m. Select the column with "O" in it (press the number 4 button).

n. Select "O" (press the number 3 button).

o. Select the column with "N" in it (press the number 4 button).

p. Select "N" (press the number 2 button).

q. Press the Cont/Pause button.

r. Press the zero button.

s. Select "Page down" (press the number 2 button).

t. For all results, the result divided by the standard deviation should be less than five (5). If it is not, acquire new background data are discussed in Section E of this section.

6. You now need to modify the scanning times for site specific calibration.

   a. Select "modify" (press the number 1 button).

   b. Select "Mod" (press the number 3 button).

   c. Enter 40 and press the Cont/Pause button.
   d. Select "Down" (press the number 2 button).

   e. Select "Mod" (press the number 3 button).

   f. Enter 10 and press the Cont/Pause button.

   g. Select "Down" (press the number 2 button).

   h. Select "Mod" (press the number 3 button).

   i. Enter 10 and press the Cont/Pause button.

   j. Select "Exit" (press the number 6 button).

7. You are now ready to analyze the site-specific standards.

   a. Place one of the site-specific standards over the source window.

   b. Close the shield cup lid.

   c. Press the Cont/Pause button.

   d. You will now see the label screen.

   e. Select the column with the first letter or number of your standard name (press the appropriate number button).

   f. Continue this process for the entire standard label.

   g. Press the Cont/Pause button.

h. Press the zero button.

i. Select "Page down" (press the number 2 button).

j. Note the value for lead (Pb) or whatever elements for which you are analyzing the samples.

k. Repeat steps c-j for the standard two more times. Each standard should be analyzed in triplicate.

l. The average of the three values found for the standard should be within ± 20% of the known value of the standard. If it is now, perform an energy calibration. The procedures for an energy calibration are discussed in Section D of this section.

m. Repeat steps a-l for all other site-specific standards.

The XRF is now ready to be used.

D. Energy Calibration

1. You are now at the Main menu. Select measure (press the number 1 button).
2. Select "Options" (press the number 5 button).
3. Select "Energy calibration" (press the number 1 button).
4. The XRF will then say "Measure Safety Cover".
5. Put the safety cover on the probe.
6. Select "Proceed" (press the number 1 button).
7. The XRF will return to the analysis screen when the energy calibration is complete.

E. Background Data Acquisition

1. You are now at the Main menu. Select measure (press the number 1 button).
2. Select "Options" (press the number 5 button).
3. Select "Acquire background data" (press the number 2 button).
4. The XRF will then say "Measure Quartz".
5. Put the quartz standard on the probe.

6. Select "Proceed" (press the number 1 button).

7. The XRF will return to the analysis screen complete and give further instructions. Follow these instructions until acquisition is complete.

XIII. Sample Preparation

A. In-situ Samples

1. Clear the soil of all vegetation.
2. Clear the soil of any debris that may puncture the aperture window.
3. Tamp the soil to ensure it is flat and free of voids.

B. Collected Samples

1. Dry the samples in an oven or microwave oven.
2. Grind the samples into a fine powder, removing any large rocks or debris.
3. Homogenize the sample to ensure consistency.
4. Place the soil into an XRF soil cup, cover with film and seal.

XIV. Analytical Measurement

A. Instrument Set-up

1. Place the electronics portion of the XRF on a flat surface, adjusting the handle to be used as a stand.

2. Connect the Electronics portion to the probe using the interconnecting cable.

   a. When inserting the cable into the probe and electronics portion, pull back metal cover on end of the cable, align the red dot on the cable with the grove on the insertion point and finally gently insert the cable until you hear a soft "click".

3. Remove the safety cover from the probe.

B. Turn on procedures

1. Turn on the unit.
   a. Press the "On" button.

   b. You will then receive the prompt, "Is 0:00:00 the correct time?". If it is the correct time, press "yes" (the number 1 button). If it is now the correct time, press "no" (the number 2 button). The XRF will then instruct you on how to reset the time.

   c. You will then receive the prompt, "Is 0:00:00 the correct date?" If it is the correct date, press "yes" (the number 1 button). If it is not the correct date, press "no" (the number 2 button). The XRF will then instruct you on how to reset the date.

d. Allow the XRF to warm-up for at least 10 minutes.

C. Field use

1. You are now at the main menu. Select measure (press the number 1 button).

2. You now may need to modify the scanning time.

    a. Select "modify" (press the number 1 button).

    b. Select "Mod" (press the number 3 button).

    c. Enter 40 and press the Cont/Pause button.

    d. Select "Down" (press the number 2 button).

    e. Select "Mod" (press the number 3 button).

    f. Enter 10 and press the Cont/Pause button.

    g. Select "Down" (press the number 2 button).

    h. Select "Mod" (press the number 3 button).

    i. Enter 10 and press the Cont/Pause button.

    j. Select "Exit" (press the number 6 button).

3. You are now ready for analysis

    a. Place one of the samples over the source window or place the probe on the area to be analyzed making sure the window is not punctured.

    b. Close the shield cup lid if applicable.

    c. Press the Cont/Pause button.

    d. You will now see the label screen.

    e. Select the column with the first letter or number of your sample name (press the appropriate number button).

    f. Continue this process for the entire sample label.

    g. Press the Cont/Pause button.

h. Press the zero button.

i. Select "Page down" (press the number 2 button).

j. Note the value for lead (Pb) or whatever elements for which you are analyzing the samples.

k. Repeat steps c-j for the standard two more times. Each standard should be analyzed in triplicate.

XV. Data Treatment

A. The result at each sample location is the average of three reading taken at the location.

Result = (Reading 1 + Reading 2 + Reading 3) / 3

B. All readings must be greater than three times the XRF calculated standard deviation in order to be considered valid.

Reading > 3 * Standard deviation

If the above level is not achieved increase the scan time until it is achieved.

XVI. Data Deliverables

-The following documents are available to the client upon request:

A. A summary of initial, ongoing and end of analysis calibration results. This should include each reading, the average of the three readings for each sit-specific standard and the percent difference between the result and the laboratory determined value.

B. A logbook detailing the following:

1. Weather conditions
2. Sampler/s
3. Date of analysis
4. Time of each analysis
5. Location of each analysis
6. Sample preparations required
7. Results of each analysis
8. Any problems encountered and corrective actions taken
9. List date of XRF purchase, latest calibration, leak test and source replacement

C. A printout of all results saved on the XRF and downloaded to a PC. This will be downloaded and formatted in EXCEL and will include sample number, date taken and value in ppm.

D. A summary of all QC required. This will be determined on a site specific basis.

XVII. Quality Control Requirements

A. The quality control requirements for the use of the XRF are determined on a site specific basis. These are to be addressed in the site work plan and quality control plan. The exact requirements will vary depending on the use of the XRF on the site. However, all plans should require instrument calibration prior to and after XRF usage.

XVIII. References

A. Spectrace 9000 Analyzer Manual
TN Technologies Inc.
1992, 1993 and 1994

B. Quality Assurance Technical Information Bulletin
US Environmental Protection Agency
Vol. 1, No. 4
May 1991